

January 1957

Pages 1-40

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 73 Number 1

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NEW COLOUR INDEX

See important announcement
on pages xix to xxii in
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THE SOCIETY OF DYERS AND COLOURISTS
19 PICCADILLY BRADFORD 1 YORKSHIRE

Notice to Authors of Papers

The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

- | | |
|---|--|
| The Chemistry of Esters of Leuco Vat Dyes—II | <i>S. Ainsworth and A. Johnson</i> |
| Measurement of Diffusion Coefficients of
Azoic Coupling Components in Cellulose | <i>A. S. Dunn</i> |
| A Method of Isolation of the Cuticle
Sheath from Wool Fibres | <i>R. L. Elliott and J. B. Roberts</i> |
| Light Fastness Assessments of Dyed Textiles
and their Bearing upon the Mechanism of Fading | <i>C. H. Giles</i> |
| Precise Measurement of Fading on a
Time-Intensity Basis | <i>J. S. Mudd</i> |
| Alginate Hessian | <i>E. N. Woodward et al.</i> |

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FORTHCOMING MEETINGS OF THE SOCIETY

Wednesday, 23rd January 1957

MIDLANDS SECTION. *Problems in the Processing of Rayon Blends*. R. C. Cheetham, Esq., A.M.C.T., F.R.I.C., and H. D. Edwards, Esq., Ph.D., A.R.I.C. College of Technology, **Leicester**. 7 p.m.

Tuesday, 29th January 1957

BRADFORD JUNIOR BRANCH. *The Work of a Colourist in a Man-made Fibres Dyehouse*. J. T. Lynes, Esq. (Courtaulds Ltd.). Technical College, **Bradford**. 7.15 p.m.

Thursday, 31st January 1957

WEST RIDING SECTION. Discussion, *Detergency*. Panel—R. S. Hartley, Esq., A.R.I.C. (Wool Industries Research Association), G. H. Rostron, Esq., A.R.I.C. (Joseph Crosfield & Sons Ltd.), R. C. Tarring, Esq., B.Sc., A.R.C.S. (Shell Chemicals Ltd.). Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

Friday, 1st February 1957

LONDON SECTION. *A Review of the Colour Fastness Requirements in Dyed Textiles*. T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C. (Courtaulds Ltd.). (Joint meeting with the London Section of the Clothing Institute.) Royal Society, Burlington House, **London W.1**. 6 p.m.

Tuesday, 5th February 1957

LEEDS JUNIOR BRANCH. *Supply, Treatment and Disposal of Water in Relation to the Dyehouse*. R. W. Richardson, Esq., B.Sc., Ph.D., A.R.I.C. (Courtaulds Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds 2**. 3.30 p.m.

MANCHESTER JUNIOR BRANCH. "... and inwardly digest". A Dissertation on the difficulty of assimilating Technical literature. S. Burgess, Esq., A.M.C.T., F.S.D.C. (Courtaulds Ltd., Droylsden Dyehouse). Manchester College of Science and Technology, **Manchester**. 7 p.m.

Thursday, 7th February 1957

THE GEORGE DOUGLAS LECTURE. *Dyeing*. G. S. J. White, Esq., M.A., F.T.I., F.S.D.C. The Midland Hotel, **Manchester**. 7.15 p.m.

Tuesday, 12th February 1957

SCOTTISH SECTION. *Reactive Dyes—A New Approach to Cellulose Dyeing*. R. W. Speke, Esq., B.Sc. (I.C.I. Ltd.). St. Enoch Hotel, **Glasgow**. 7.15 p.m.

Wednesday, 13th February 1957

NORTHERN IRELAND SECTION. *Fifty Years Dyeing Linen in Ireland*. N. McCraith, Esq., B.Sc. (Joint Meeting with Textile Institute.) Thompson's Restaurant, Donegall Place, **Belfast**. 7.30 p.m.

BRADFORD JUNIOR BRANCH. *Finishing of Garments and Fabrics made from Bulk "Terylene" Yarns and Filaments*. G. McLewy, Esq., B.Sc., Ph.D., A.R.I.C. (I.C.I. Ltd. Terylene Council). Technical College, **Bradford**. 7.15 p.m.

Thursday, 14th February 1957

WEST RIDING SECTION. *Applications of Colour Physics to Textiles*. J. C. Guthrie, Esq., M.Sc., F.Inst.P., F.T.I., P. H. Oliver, Esq., B.Sc., Grad.Inst.P. (Courtaulds Ltd.). Hotel Metropole, King Street, **Leeds**. 7.30 p.m.

Friday, 15th February 1957

MANCHESTER SECTION. *Problems in the Wet Processing of Rayon Blends*. H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Textile Institute, 10 Blackfriars Street, **Manchester**. 7 p.m.

Tuesday, 19th February 1957

HUDDERSFIELD SECTION. *Petroleum Chemicals in the Textile Industry*. H. Bratherton, Esq., A.M.C.T., A.T.I. Silvios Cafe, Westgate, **Huddersfield**. 7.30 p.m.

Wednesday, 20th February 1957

MIDLANDS SECTION. *Basic Mechanisms of Winch Processing*. R. H. Waddington, Esq., B.Sc. Albert Hall Institute, **Nottingham**. 7 p.m.

Thursday, 28th February 1957

WEST RIDING SECTION. *The Applications of Fluid Beds to Dyeing*. J. Wilson, Esq., M.C., M.Sc. and F. G. Audas, Esq. (British Rayon Research Association). Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

Tuesday, 5th March 1957

LEEDS JUNIOR BRANCH. *Reactive Dyes. A New Approach to Cellulose Dyeing*. C. D. Weston, Esq. (Imperial Chemical Industries Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds 2**. 3.30 p.m.

MANCHESTER JUNIOR BRANCH. *The Principles and Practice of Package Dyeing*. K. Limbert, Esq. (The Longclose Engineering Co. Ltd., Leeds). Manchester College of Science and Technology, **Manchester**. 7 p.m.

Thursday, 7th March 1957

BRADFORD JUNIOR BRANCH. *Flame Proofing*. J. R. W. Perfect, Esq. (Bradford Dyers' Association Ltd.). Technical College, **Bradford**. 7.15 p.m.

Friday, 8th March 1957

LONDON SECTION. *Soiling of Natural and Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). Royal Society, Burlington House, **London W.1**. 6 p.m.

Tuesday, 12th March 1957

SCOTTISH SECTION. *Recent Developments in Wool Dyeing*. G. H. Lister, Esq., Ph.D., B.Sc. (Sandoz Products Ltd.). St. Enoch Hotel, **Glasgow**. 7.15 p.m.

Wednesday, 13th March 1957

NORTHERN IRELAND SECTION. *Problems in Wet Processing of Rayon Blends*. H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Thompson's Restaurant, Donegall Place, **Belfast**. 7.30 p.m.

Thursday, 14th March 1957

WEST RIDING SECTION. *The Dyeing of Acrylic Fibres*. K. Meldrum, Esq., B.Sc., A.R.I.C. (Courtaulds Ltd.). Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

Tuesday, 19th March 1957

HUDDERSFIELD SECTION. Annual General Meeting. Silvios Cafe, Westgate, **Huddersfield**. 7.30 p.m.

Wednesday, 20th March 1957

MIDLANDS SECTION. *Developments in Sizing as they affect the Dyer and Finisher*. J. H. MacGregor, Esq., Ph.D., F.R.I.C., and E. France, Esq., A.R.C.S., A.M.C.T. (Joint with the British Association of Chemists.) Midland Hotel, **Derby**. 7 p.m.

Friday, 22nd March 1957

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, **Bradford**.

Saturday, 23rd March 1957

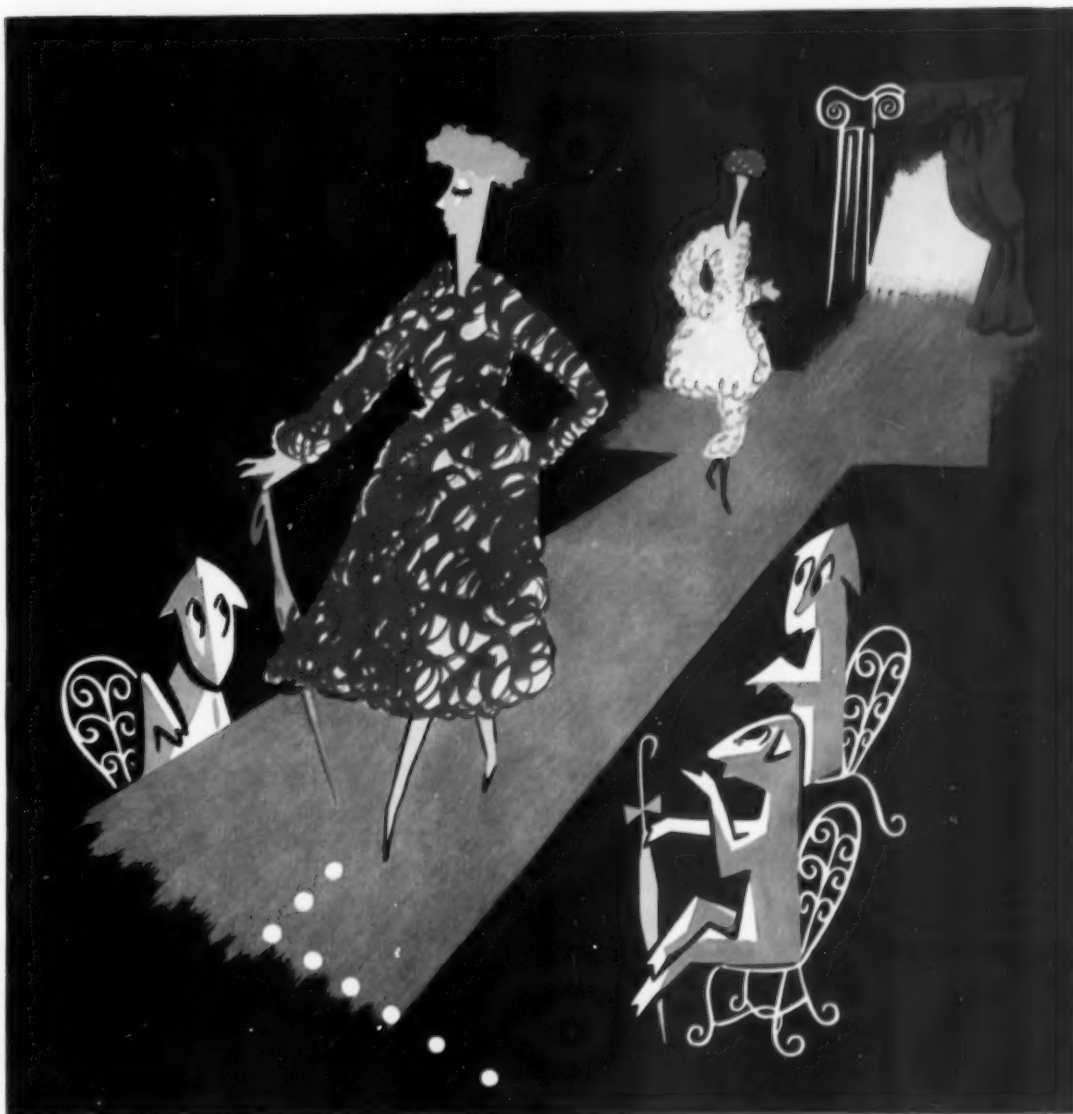
NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Woodbourne House Hotel, **Belfast**. Details later.

Thursday, 28th March 1957

MIDLANDS SECTION. *The Soiling of Natural and Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Joint with the Textile Institute.) Carpet Trades Canteen, **Kidderminster**. 7.30 p.m.

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

continued on page XXXV



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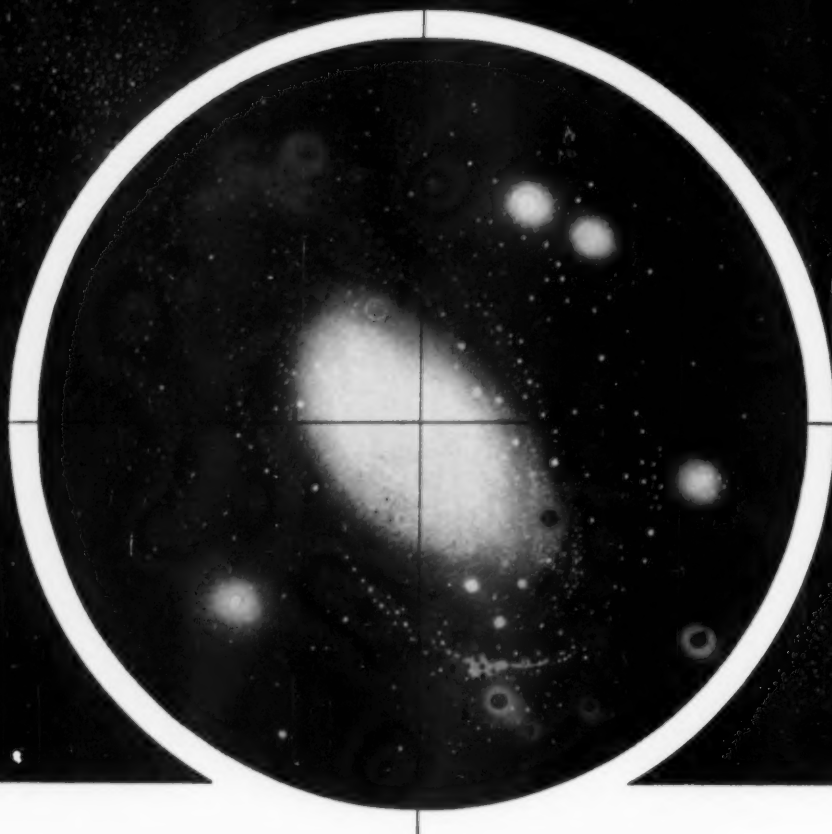
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OF THE

Society of Dyers and Colourists

Volume 73 Number 1

JANUARY 1957

Issued Monthly

OFFICIAL NOTICES

OBJECTS OF THE SOCIETY OF DYERS AND COLOURISTS

To promote the advancement of science and technology, especially in the theory and practice of the tinctorial arts.

To provide means for the wider dissemination and interchange of knowledge concerning the science and technology of colour and colouring matters, of their methods of application, and of the materials to which they may be applied.

To encourage education and research in any or all of the above subjects.

And for the furtherance of these objects—

To hold meetings for the reading of papers, for lectures, and for discussions.

To publish a *Journal*.

And to do all such other things as may be conducive to the attainment of these objects.

ELECTION OF OFFICERS AND MEMBERS OF COUNCIL

Under the revised Bye-laws which are now operative, Officers and Members of Council—other than the President and President-elect, who are nominated by Council and elected at the Annual General Meeting—are to be elected by a Postal Ballot of the members.

The attention of members is drawn to Bye-laws No.

12-21 inclusive and 22-26 inclusive, which relate to Officers and Members of Council respectively.

Nominations to be valid must be received by the Honorary Secretary of the Society at least six weeks prior to the date of the Annual General Meeting, which is on 29th March 1957.

MEMBERS AND JUNIOR MEMBERS

Persons desirous of joining the Society as Ordinary Members or Junior Members may obtain Application Forms from the Secretary or from the Honorary Secretary of any Section of the Society. Applications must be proposed and seconded by members to whom the applicants are known personally. The rates of annual subscription are as follows—

(i) During the period of "full-time national service" the annual subscription to be waived entirely. Such members will be asked whether they wish to receive the *Journal*.

(ii) Ordinary Members between the ages of 21 and 25 years who are students, i.e. who are pursuing an accepted course, full or part time, at a recognised

technical college or university and vouched for by the head of their department or other responsible person—15s. 0d.

(iii) Other Ordinary Members between the ages of 21 and 25 years—£2 2s. 0d.

(iv) Members having had forty years' continuous membership of the Society to be given the option of paying half the current annual subscription (vi) for Ordinary Members.

(v) Members having had fifty years' continuous membership of the Society—the annual subscription to be waived entirely.

(vi) All other Ordinary Members—£3 3s. 0d.

(vii) All other Junior Members—15s. 0d.

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The Society confers diplomas in tinctorial technology on suitably qualified members. These distinctions are in two grades—the Associateship and the Fellowship—and confer the right to use the title Associate of the Society of Dyers and Colourists (A.S.D.C.) or Fellow of the Society of Dyers and Colourists (F.S.D.C.). To obtain the Associateship candidates are required to pass the proscribed examinations and to furnish evidence of satisfactory general education and of training and experience in at least one branch of the manufacture or application of

colouring matters. The Fellowship is conferred on senior members who have attained high standing in the knowledge and practice of tinctorial technology. Students who intend to take the Society's examination are strongly recommended to become Registered Students, so that their studies and preparation may be effectively directed.

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Members are reminded that under Bye-law 41 the *Journal* will not be forwarded to those who have not paid their subscription by 30th June 1957.

Members residing abroad are particularly requested to inform the Secretary by separate post when sending their subscriptions by Money Order.

Communications on any subject related to the objects of the Society, especially such as are of an original character, are invited for consideration for publication in the *Journal*. Such Communications in the first instance should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1, Yorkshire.

The JOURNAL—contd.

General communications, including inquiries or orders for advertisements, should be addressed to the Offices of the Society, to which address all remittances should be sent.

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I—The Society is prepared to receive from members and others and to keep as deposits, Sealed Communications dealing with any subject relating to the theory or practice of the Dyeing, Printing, and kindred industries.

II—Every deposit must bear on the cover a title for classification, the author's name, the date, and must be secured by a distinctive seal.

III—The deposit should be written in English, and, if it deals with machinery, be accompanied by sketches or drawings, or, in the case of dyeing or printing processes, by patterns.

IV—Every deposit will, unopened, be signed and sealed by the Honorary Secretary of the Society, immediately after receipt, and countersigned by another member of the Council.

V—The deposits will be numbered and entered into a

special register, and an official receipt will be sent to the author.

VI—No charge will be made for registration and deposit.

VII—The deposits will be kept by the Society for a period of seven years. At the end of this period they will be opened and the contents read before the next meeting of the Council of the Society, and the contents, or an abstract thereof, may be published in the *Journal* of the Society at the discretion of the Publications Committee.

VIII—The author shall have the right to recall his deposit unopened at any time within the said seven years, or he may, at any time, order it to be opened and read before one of the Society's meetings. Such instructions must be accompanied by the official receipt.

IX—The Society will take every reasonable care of the deposits, but cannot be held responsible for their loss.

MEDALS AND AWARDS

HONORARY MEMBERS OF THE SOCIETY

1886-1915 *Sir H. E. Roscoe, M.P.
1902-1926 *Dr. F. H. Bowman
1908-1916 *Prof. C. Graebe
1908-1915 *Prof. C. Liebermann
1911-1916 *Prof. Adolf von Baeyer
1914-1924 *Count Hilaire de Chardonnet
1917-1941 *Prof. Arthur G. Green
1919-1938 *R. Vidal
1921-1930 *Horace A. Lowe
1923-1935 *Charles F. Cross
1925-1927 *Maurice Prud'homme
1927-1930 *Ernest Hickson
1928-1939 *Dr. R. E. Schmidt
1931-1933 *Dr. Alfred Réé

1932-1940 *Prof. W. M. Gardner
1934-1947 *George Douglas
1934-1946 *H. Grandage
1934-1940 *Christopher Rawson
1941-1948 *Prof. E. C. C. Baly, C.B.E.
1944-1946 *Prof. F. M. Rowe
1946 *James S. Ridsdale
1947 Dr. C. J. T. Cronshaw
1948-1956 *H. Jennison, M.C.
1950-1953 *George E. Holden, C.B.E.
1953 John Barritt
1954 Fred Scholefield
Dr. Croyden Meredith Whittaker
1955 Sir Robert Robinson, O.M., Nobel Laureate

The following have been *ex officio* Honorary Members of the Society, the later dates indicating termination or change in title of the office—

1886-1920 The Worshipful Master of the Dyers' Company
1920- The Prime Warden of the Worshipful Company of Dyers

1886-1900 The President, Bradford Technical College
1900-1905 *W. E. B. Priestley, Chairman of the Technical Instruction Committee of the Bradford City Council

*Deceased

THE PERKIN MEDAL

The Perkin Medal was modelled by the late F. W. Pomeroy, R.A., for the Society. It is an excellent presentation of the head of Sir William Perkin, the founder of the coal-tar colour industry, and President of the Society in 1907.

This medal is awarded for discoveries or work of outstanding importance in connection with the tinctorial arts.

LIST OF RECIPIENTS

1908 Professors Graebe and Liebermann. "Synthesis of Alizarin."
1911 Prof. Adolf von Baeyer. "Synthesis of Indigo."
1914 Comte Hilaire de Chardonnet. "Artificial Silk."
1917 Prof. Arthur G. Green. "Primuline."
1919 R. Vidal. "Sulphur Black."
1921 Horace Lowe. "Permanent Lustre on Cotton."
1923 Chas. F. Cross. "Discovery of Viscose."
1925 M. Prud'homme. "Aniline Black and Alizarin Blue."
1928 Dr. Robert E. Schmidt. "For Epoch-making Discoveries of Anthraquinone Derivatives and Dyestuffs therefrom."
1938 Dr. H. Dreyfus. "For Discoveries and Work of Outstanding Importance in Connection with the Development of the Cellulose Acetate Rayon Industry in England."
1938 J. Baddiley. "In recognition of his National Services for the Renaissance of the British Dyestuffs Industry through Many Important Investigations in the Field of Colour Chemistry Conducted or Directed by him."
1950 Prof. J. B. Speakman. "In recognition of his Outstanding Contributions to the Science and Technology of Textiles."
1954 Dr. Arthur Zitscher. "For his Work leading to the Discovery of the New Class of Azoic Dyes, based on the Arylamides of o-Hydroxycarboxylic Acids."

1956 Dr. Wallace H. Carothers (posthumous award). "For the Discovery of Nylon."
John R. Whinfield. "For the Discovery of Terylene."





THE MEDAL OF THE SOCIETY OF DYERS AND COLOURISTS

This Medal was instituted by the Society in 1908. Of the competitive designs submitted, that of Mr. Edgar Lockwood was selected. The design indicates that the work of the dyer (centre figure) is both a science (right background) and an art (left foreground).

1908-1927 The Medal was occasionally awarded as a recognition of work of exceptional merit carried out under the Society's Research Scheme.

From 1928 The Medal was awarded as a recognition of exceptional services (a) to the Society or (b) in the interests of the Tinctorial and Allied Industries.

LIST OF RECIPIENTS

- 1908 J. B. Fothergill (Bronze Medal). "Treatment of Cotton to Cause it to Resist Direct Dyeing Colour." 1947 Fred Smith (Gold Medal). "For Exceptional Services to the Society over a period of thirty-seven years comprising Chairman, West Riding Section; Member of Council and of many important Committees."
- 1912 J. H. Garner (Silver Medal). "Treatment of Effluents from Dyehouses and Textile Factories." F. Scholefield (Gold Medal). "In recognition of Exceptional Services to the Society in the advancement of Tinctorial Technology both in theory and practice."
- 1928 Ernest Hickson (Gold Medal). "Exceptional Services rendered to the Society as Chairman of the Publications Committee 1897-1925, and Chairman of Colour Index Committee." C. Schardt (Silver Medal). "In recognition of his Valuable Services to the Society over a period of twenty-four years including Chairman, Vice-Chairman, and member, of the Midlands Section Committee."
- 1930 Arthur Silverwood (Gold Medal). "Exceptional Services rendered to the Society as Honorary Secretary 1913-1930." 1948 G. G. Hopkinson (Gold Medal). "For Valuable Services rendered to the Society and to the Dyeing Industry."
- 1933 Prof. Walter M. Gardner (Gold Medal). "Distinguished Services as Editor of the Society's Journal 1900 to 1932." D. B. F. McAndrew (Silver Medal). "For Valuable Services rendered to the Society as Honorary Secretary and Committee Member of the Scottish Section 1935-1947."
- 1934 Prof. F. M. Rowe (Gold Medal). "Exceptional Services to the Society and to the Tinctorial Industries as Editor of the Colour Index 1924 and the 'Supplement' 1928." C. O. Clark (Silver Medal). "For Valuable Services rendered to the Society from 1923 to 1948."
- L. A. Lantz (Chairman), H. H. Bowen, P. W. Cunliffe, R. S. Horsfall, Prof. B. A. McSwiney, C. C. N. Vaas, C. M. Whittaker, S. G. Barker (Silver Medals). "Exceptional Services to the Society as Members of the Standardisation of Fastness Executive Committee." 1949 S. M. Neale (Gold Medal). "For his Pioneer Work in the Application of the Methods of Physical Chemistry to the Elucidation of the Phenomena of Dyeing, more particularly of Cellulosic Materials with Substantive Dyes."
- 1936 W. A. Edwards (Silver Medal). "Valuable Services rendered to the Society as Honorary Secretary of the Midlands Section since its inception in 1919 to 1935." 1950 P. W. Cunliffe (Gold Medal). "For Outstanding Services to the Society and to the Tinctorial and Allied Industries."
- 1937 R. Ritchie (Silver Medal). "Devoted and Valuable Services as Honorary Secretary of the Scottish Section for eighteen years." H. Foster (Gold Medal). "For Outstanding Services to the Society."
- 1940 F. L. Goodall (Gold Medal). "Valuable Services to the Tinctorial Industries by his work on the Theory and Practice of Wool Dyeing." L. A. Lantz (Bar attached to Silver Medal previously awarded). "For Exceptional Services to the Society and to the Tinctorial and Allied Industries in connection with Fastness Tests."
- 1943 C. M. Whittaker (Gold Medal). "In recognition of Exceptional Services in promoting Scientific and Technical Knowledge amongst Textile Colourists, and for Sustained and Outstanding Service to the Society." 1951 W. Kilby (Gold Medal). "For Work on the Development of a Molten Metal Process of Continuous Dyeing."
- 1946 H. H. Bowen (Gold Medal). "For Outstanding Services to the Society and in recognition of his Chairmanship of the Publications Committee for a period of twenty years." 1953 H. A. Turner (Gold Medal). "For his Valuable Researches contributed to the Tinctorial Industries."
- H. H. Hodgson (Gold Medal). "For Outstanding Services to the Society and for his series of Sustained Experimental and Theoretical Contributions to those chapters of Organic Chemistry which are the essential scientific background to the Dyestuffs Industry." A. W. Carpenter (Silver Medal). "For Valuable Services to the Society."
- E. Race (Silver Medal). "For his Valued Services to the Society as Joint Author of thirteen papers published in the Society's Journal." Miss M. Forbes (Bronze Medal). "For Valuable Services to the Society."
- H. Turner (Silver Medal). "In recognition of his Valuable Services to the Society and of his twenty-two years' service as Honorary Secretary of the Huddersfield Section." H. R. Hadfield (Bronze Medal). "For Valuable Services to the Society."
- Mrs. E. Cummings (née Levin) (Silver Medal). "For her Valued Services to the Society; by her assistance rendered in the preparation of the first Colour Index; by her Joint Authorship of a number of 1954 G. S. J. White (Gold Medal). "For Exceptional Services to the Society and to the Tinctorial and Allied Industries." C. C. Wilcock (Silver Medal). "For Exceptional Services to the Society and to the Tinctorial and Allied Industries."
- 1955 J. Porter (Silver Medal). "For Outstanding Services to the Society, notably in the Inauguration and the Development of the Northern Ireland Section."

1956 P. W. Cunliffe (Bar to Gold Medal). "For Continued Valuable Services to the Society as Chairman of the Fastness Tests Co-ordinating Committee."

H. W. Ellis (Silver Medal). "For Valuable Services to the Society as Honorary Secretary of the London Section 1938-1954."

J. G. Grundy (Silver Medal). "For Valuable Services to the Society as Chairman of the Washing Fastness Subcommittee."

K. McLaren (Silver Medal). "For Valuable Services to the Society as Honorary Secretary of the Fastness Tests Co-ordinating Committee."

M. E. Probert (Silver Medal). "For Valuable Services to the Society as Chairman of the Bleaching Fastness Subcommittee."

J. V. Summersgill (Silver Medal). "For Valuable Services to the Society as Honorary Secretary of the Publications Committee from 1945 to date."

E. Wilson (Silver Medal). "For Valuable Services to the Society as Chairman of the Alkaline Milling, Burnt-gas Fumes, and Perspiration Fastness Subcommittees."

THE WORSHIPFUL COMPANY OF DYERS RESEARCH MEDAL

The Medal represents the Arms of the Worshipful Company of Dyers of the City of London, which were granted in 1471. The following is a brief description—

Arms—Sable, a chevron engrailed argent, between three bags of madder of the last, corded or.

Crest—On a wreath three sprigs of the gaintree erect vert, fructed gules.

Supporters—Two leopards rampant gardant argent, spotted with various colours; fire issuing from their ears and mouth proper, both ducally crowned or.

Motto—Da Gloriam Deo.

1—The Dyers' Company offer annually a Gold Medal called "The Worshipful Company of Dyers Research Medal", the award of which is open to the Authors of papers embodying the results of scientific research or technical investigation connected with the tinctorial arts submitted to the Society of Dyers and Colourists, and published in the *Journal* of such Society during the twelve months ending on the 30th June in the year for which the Medal is granted, and, in the special circumstance provided for by Rule 6, during the twelve months ending on the 30th June in the year previous to that for which the Medal is granted. If a paper shall be published in two or more parts, then for the purpose of the award of the Medal, all the parts together shall be treated as a paper published in the year in which the final part is published.

2—The Medal will not be awarded to the same person on more than one occasion.

3—The Society of Dyers and Colourists shall consider the papers available for the Medal and advise the Company as to the merits thereof, and if, in the judgment of the Society, none of the papers is of sufficient merit, the Society may recommend that the Medal be not awarded.

4—The Dyers' Company will award the Medal either to the Author of the paper which, in all the circumstances, appears to the Company to show the greatest merit, or, in the event of such paper being the work of an Author who has already been awarded the Medal, to the Author of the paper next in order of merit who has not already been awarded the Medal, and may, if the Company so thinks fit, refrain from making any award.

5—In the event of a paper being the work of two or more persons, the Author shall be taken to be that one of them whose work in the opinion of the Society of Dyers and Colourists has most substantially contributed to the merit of the scientific research or technical investigation embodied in such paper.

6—In the event of the Author of a paper of sufficient merit published in the *Journal* of the Society of Dyers and Colourists during any twelve months for which the Medal is awarded being unsuccessful in obtaining the award of the Medal for that period, the Society of Dyers and Colourists may, in their discretion, consider such paper for adjudication with the papers available for the award of the Medal for the next succeeding twelve months.

LIST OF RECIPIENTS

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|---------|---|---------|--|
| 1908 | Prof. E. Knecht. "A Means of Estimating the Degree of Mercerisation in Cotton Yarns." | 1917-18 | L. G. Radcliffe. "The Sulphonation of Fixed Oils." |
| 1909 | Prof. Arthur G. Green. "The Chemical Technology of Aniline Black." | 1918-19 | Chas. F. Cross (Diplomas presented to M. C. Lamb and C. V. Greenwood as co-authors). "Colloidal Tannin Compounds and their Applications." |
| 1910-11 | R. L. Taylor. "The Action of Carbon Dioxide and of Air on Bleaching Powder." | 1919-20 | A. E. Everest (Diploma presented to A. J. Hall as co-author). "The Tinctorial Properties of some Anthocyanins and certain Related Compounds." |
| 1911-12 | W. Harrison. "The Electrical Theory of Dyeing." | 1920-21 | Prof. G. T. Morgan. "The Co-ordination Theory of Valency in Relation to Adjective Dyeing." |
| 1912-13 | S. H. Higgins. "Observations on the Bleaching of Cotton" and "The Action of Neutral Salts on Bleaching Solutions." | 1921-22 | S. Judd Lewis. "On the Fluorescence of Cellulose and its Derivatives." |
| 1913-14 | W. Johnson (in conjunction with Prof. Arthur G. Green, who had already been awarded the Medal). "The Constitution of Aged and of Bichromate Aniline Blacks" | 1922-23 | Prof. Arthur G. Green (Bar attached to Medal previously awarded), and K. H. Saunders. "The Ionamines—A New Class of Dyestuffs for Cellulose Acetate Silk." |
| 1914-15 | Morris Fort. "The Mechanism of the Acid Dye-bath." | 1923-24 | S. Judd Lewis (Bar attached to Medal previously awarded). "The Quantitative Determination of the Fluorescent Power of Cellulose and its Derivatives." |
| 1915-16 | James R. Hannay. "The Interaction between Metallic Copper and certain Dyes of the Thiazine, Oxazine and Azine Series." | | |
| 1916-17 | Prof. H. M. Dawson. "The Phenomena of Acid Catalysis and the Theory of Acids." | | |



- 1924-25 Prof. F. M. Rowe (with Diploma to Miss E. Levin, as co-author). "The Identification of Azo Colours on the Fibre and of Azo Pigments in Substance."
- 1925-26 H. H. Hodgson. "Behaviour of the Sulphides of Sodium in Aqueous and Alcoholic Media", and "The Action of Sulphur on the Monochlor-anilines."
- 1926-27 Prof. F. M. Rowe (Bar attached to Medal previously awarded) and Diplomas awarded to collaborators—Miss E. Levin, A. C. Burns, J. S. H. Davies, and W. Tepper. "A New Reaction of Certain Diazosulphonates derived from β -Naphthol-1-sulphonic acid, leading to the preparation of Phthalazine, Phthalazone and Phthalimidine Derivatives."
- 1927-28 No award.
- 1928-29 F. Scholefield (with diplomas to Miss E. Hibbert and C. K. Patel as co-authors). "The Action of Light on Dyed Colours."
- 1929-30 H. H. Hodgson (Bar attached to Medal previously awarded). "Colour and Constitution from the Standpoint of Recent Electronic Theory."
- 1930-31 Prof. F. M. Rowe (Second Bar attached to Medal previously awarded), and Diplomas awarded to S. Ueno and F. H. Jowett as collaborators. "Insoluble Azo Colours on the Fibre and Action of Boiling Caustic Soda thereon."
- 1931-32 No award.
- 1932-33 Two awards—W. T. Astbury. "The X-Ray Interpretation of Fibre Structure." J. B. Speakman. "The Structure of the Wool Fibre; its Relation to the Dyeing and Finishing Processes of the Wool Textile Trade."
- 1933-34 No award.
- 1934-35 H. A. Turner, and Diplomas awarded to G. M. Nabar and F. Scholefield, as co-authors. "The effect of Reduced Vat Dyes upon the Hypochlorite Oxidation of Cellulose."
- 1935-36 Prof. F. M. Rowe (Third Bar attached to Medal previously awarded), and Diplomas awarded to C. H. Giles, R. L. M. Allen, W. G. Dangerfield, and Glyn Owen, as collaborators. "Decomposition of Azo Dyes by Acids, Caustic Alkalies, and Reducing Agents."
- 1936-37 J. B. Speakman (Bar attached to Medal previously awarded), and Diplomas awarded to C. B. Whewell and J. L. Stoves, as collaborators. "The Reactivity of the Sulphur Linkage in Animal Fibres."
- 1937-38 Prof. F. M. Rowe (Fourth Bar attached to Medal previously awarded), and J. B. Speakman (Second Bar attached to Medal previously awarded), and Diplomas awarded to E. Race and T. Vickerstaff, as collaborators. "The Uneven Dyeing of Wool with Acid and Chrome Dyes. Part I—The Reasons for the Uneven Dyeing Properties of Wool Staples and of Cloth Damaged by Exposure, and Part II—A Method for Correcting the Uneven Dyeing Properties of Wool Staples and of Cloth Damaged by Exposure."
- 1938-39 No award.
- 1939-40 T. H. Morton, as senior author of the paper on "Application of Vat Dyes to Viscose Rayon", by J. Boulton and T. H. Morton.
- 1940-41 No award.
- 1941-42 T. Vickerstaff, as senior author of the paper on "The Dyeing of Cellulose Acetate Rayon with Dispersed Dyes", by T. Vickerstaff and E. Waters.
- 1942-43 No award.
- 1943-44 J. Boulton. "The Importance of Dyeing Rate—an Interpretation for the Practical Dyer of Recent Research on Direct Dyeing."
- 1944-45 No award.
- 1945-46 No award.
- 1946-47 H. Phillips, as senior author of the paper on "The Chemical Constitution and Physical Properties of Bisulphited Wool", by E. G. H. Carter, W. R. Middlebrook, and H. Phillips.
- 1947-48 J. M. Preston. "Some Factors affecting the Dyeing of Viscose" and "Some Aspects of the Drying and Heating of Textiles".
- 1948-49 H. Lindley, as major contributor to the paper "The Mechanism of Set and Supercontraction in Wool Fibres", by S. Blackburn and H. Lindley.
- 1949-50 No award.
- 1950-51 J. Crank. "The Diffusion of Direct Dyes into Cellulose. III—The Present State of the Theory and its Application".
- 1951-52 H. Hampson. "Advances in the Application of Vat Dyes to Viscose Rayon Cakes".
- 1952-53 R. H. Peters, as senior author of the paper on "The Reduction Properties of Vat Dyes", by W. J. Marshall and R. H. Peters.
- 1953-54 R. J. Hannay, as senior author of the papers "A New Method of pH Control in Dyeing and Some Observations on the Metachrome Process", by R. J. Hannay, W. H. Major, and R. Pickin, and "The Use of Hydrolysable Esters in the Control of Dyebaths", by R. J. Hannay and W. H. Major.
- 1954-55 C. H. Giles, as senior author of the papers "A Study of Certain Natural Dyes. I—The Adsorption of Brazilwood and Logwood Colouring Matters by Fibres", by F. M. Arshid, J. N. Desai, D. J. Duff, C. H. Giles, S. K. Jain, and I. R. Macneal, and "II—The Structure of the Metallic Lakes of the Brazilwood and Logwood Colouring Matters", by F. M. Arshid, R. F. Connelly, J. N. Desai, R. G. Fulton, C. H. Giles, and J. C. Kefalas.

THE WORSHIPFUL COMPANY OF FELTMAKERS RESEARCH MEDAL

The Feltmakers' Company offer annually a Gold Medal for Papers embodying the results of scientific research or technical investigation connected with the art of feltmaking and published in the *Journal* of the Society.

1945-1956 No award.

KNECHT MEMORIAL FUND

A Fund of £100 was subscribed as a Memorial to the late Professor Edmund Knecht, from the interest on which two prizes, in the form of books, are awarded annually to selected students of the Manchester College of Science and Technology and the Royal Technical College, Salford.

MERCER LECTURE

A sum of £20 per annum is being given to the Society for a limited period of years in commemoration of the centenary of the discovery of mercerisation. Suitable lecturers are invited to give a Mercer Lecture annually.

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|------|---|------|---|
| 1944 | N. G. McCulloch, B.Sc., F.S.D.C., and G. S. Hibbert, M.Sc., F.S.D.C. "Science in an Old Industry." | 1951 | D. Traill, Ph.D., F.R.I.C., F.T.I., F.S.D.C. "Some Trials by Ingenious Inquisitive Persons—Regenerated Protein Fibres." |
| 1945 | G. S. J. White, M.A., F.T.I., F.S.D.C., and T. Vickerstaff, M.Sc., Ph.D., F.S.D.C. "Colour." | 1952 | F. C. Wood, M.Sc., Ph.D., F.R.I.C., F.T.I., F.S.D.C. "Non-felting Wool and Wool Mixtures." |
| 1946 | D. Entwistle, B.Sc., A.R.I.C. "Regenerated Fibres from Natural Polymers." | 1953 | J. R. Blockey, M.Sc., F.R.I.C., and D. H. Tuck, A.L.C. "The Coloration of Leather." |
| 1947 | E. J. Bowen, M.A., D.Sc., F.R.S. "Colour and Constitution—The Absorption of Light by Chemical Compounds." | 1954 | N. W. Yelland, B.Sc., A.R.C.S., D.I.C. "Physics in the Dyeing, Printing, and Finishing Industries." |
| 1948 | F. Farrington, B.Sc., F.R.I.C., F.S.D.C. "Textile Printing." | 1955 | G. Landells, Ph.D., A.R.I.C., F.S.D.C. "Modern Resin Finishing." |
| 1949 | E. Wilson, B.A., F.S.D.C. "Some Applications of Chemistry to Textile Finishing." | 1956 | R. K. Fourness, B.Sc., F.R.I.C., F.S.D.C. "Disperse Dyes—Their Development and Application." |
| 1950 | A. B. Meggy, Ph.D., A.R.I.C., F.S.D.C. "Some Recent Developments in the Theory of Dyeing." | | |

GEORGE DOUGLAS LECTURE

A sum of £2000 was given to the Society by the Bradford Dyers' Association Ltd., the income from the investment of which is employed in providing a biennial lecture on some subject connected with the colouring or finishing of textiles.

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| 1949 | H. Levinstein, M.Sc., Ph.D., M.I.Chem.E., F.R.I.C., F.S.D.C. "George Douglas, His Times, and Some Thoughts on the Future." | 1953 | T. Holbro, Ph.D. "The Search for New Dyes in relation to Modern Developments in the Textile Field." |
| 1952 | R. Hill, Ph.D. "Synthetic Fibres in Prospect and Retrospect." | | |

COMMUNICATIONS

Separation of Some Disperse Dyes by Paper Chromatography

K. ELLIOTT and L. A. TELESZ

A method is described for the qualitative analysis of some disperse dyes on paper using petroleum ether (b.p. 65–70°C.) saturated with 98% methyl alcohol (2% water by volume). The appropriate amount of dye solution is spotted on the paper. After exposure to an atmosphere saturated with eluant vapour, the chromatograms are run overnight at 19–21°C. Details of the method and some R_f values are given in the text.

INTRODUCTION

Disperse dyes, which are mainly azo and anthraquinonoid derivatives, are the most important colouring matters for the dyeing of cellulose acetate, nylon, and Terylene, and are widely used in the hosiery industry. In the course of a series of investigations in these laboratories it was found necessary to separate mixtures of disperse dyes available only in small quantities, and our attention was turned to paper chromatography. A search of the literature revealed that relatively little information on the application of paper chromatography to this particular class of dyes had been published. References were found to work by Mitchell¹, who separated five closely related anthraquinone dyes by chromatography using paper which had been pre-impregnated by spraying with an ethereal solution of soya-bean oil (stationary phase) and

using aqueous methyl Cellosolve (20% water by volume) as eluant (mobile phase). Zahn² also succeeded in obtaining paper chromatograms of a limited number of Celliton (BASF) dyes using 60% ethyl glycol as the eluant at 15°C. Shibata, Takito, and Tanaka³, in studying the active principle in crude drugs, applied the technique of paper chromatography to the separation and identification of some naturally occurring anthraquinone pigments. They used benzine (b.p. 45–70°C.) saturated with aqueous methanol as solvent and alcoholic magnesium acetate solution for spraying the chromatograms to produce coloured spots on subsequent drying.

In a search for a simple method which would ultimately be suitable for use in a small or medium-size industrial laboratory we have found that a wide range of commercial disperse dyes of both the

TABLE I
 R_f Values calculated from Ascending Paper Chromatograms

(Eluted overnight at a mean temp. of 19–20°C. with petroleum ether (b.p. 65–70°C.) saturated with 98% methyl alcohol)

Code Letter (Fig. 1 and 2)	Dye or Dye Mixture	Colour of Band	R_f Value
(a)	Dispersol Fast Yellow G (ICI)	Yellow	0.23
(b)	*Duranol Brilliant Blue BN (ICI)	Blue	0.04
		Blue	0.95
(c)	† Dispersol Fast Red AN (ICI)	Red	0.01
		Red	0.58
(d)	Dispersol Fast Yellow G (ICI)	Red	0.01
	*Duranol Brilliant Blue BN (ICI)	Blue	0.04
	Dispersol Fast Red AN (ICI)	Yellow	0.23
		Red	0.58
		Blue	0.95
(e)	Supracet Hosiery Brown 59700 (LBH)	Red	0.02
		Yellow	0.23
		Red	0.36
		Blue	0.95
(f)	*Multamine Hosiery Brown BV (ICI)	Red	0.02
		Blue	0.05
		Yellow	0.23
		Red	0.36
		Blue	0.95
(g)	Dispersol Hosiery Brown R (ICI)	Yellow	0.09
		Blue	0.95
		Red	0.96
(h)	Seriny! Blue 2G (YDC)	Blue	0.00
	Dispersol Fast Yellow A (ICI)	Red	0.02
	Dispersol Fast Red AN (ICI)	Yellow	0.09
		Red	0.61

* In these cases a faint red band corresponding to an R_f value of 0.96 was observed when the chromatograms were viewed by transmitted light.

† The faint red band corresponding to an R_f value of 0.96 is probably due to a trace impurity, as it was not again observed on chromatograms of this dye.



FIG. 1—Ascending Chromatograms of Single Components and of a Made-up Mixture (cf. Table I)



FIG. 2—Ascending Chromatograms of Made-up and Commercial Mixtures (cf. Table I)



azo and the anthraquinonoid types could be separated and identified on paper using as eluant petroleum ether (b.p. 65–70°C.) saturated with 98% methyl alcohol. The details of the method are given below.

1. MATERIALS

Whatman No. 3 MM, a moderately fast running paper, was used. This was cut in the machine direction into sheets 20 in. \times 8 in.

The eluant was prepared by shaking in a separating funnel 480 ml. of petroleum ether (b.p. 65–70°C.) with 16 ml. of 98% methyl alcohol (containing 2% of water by volume) at 19–20°C. After standing, the mixture separated into two distinct layers, and the lower one, containing excess of alcohol, was run off and discarded. A small amount of petroleum ether was then added to make the solvent incompletely saturated with respect to aqueous alcohol. In this way the system is able to adjust itself without phase separation should the temperature during elution drop below that at which saturation of the solvent is carried out.

In the initial trials, a number of solvents, including ethylene and diethylene glycols, were used but not with entirely satisfactory results. The commercial petroleum ethers of different boiling ranges were tried, but appeared to be of little value when used alone. In the presence of the lower alcohols, and particularly methyl alcohol, a partial separation was obtained with certain dyes only. The inclusion of a small amount of water was found to enhance the resolving power of the eluant considerably.

The best results were obtained with the solvent having a boiling point of 65–70°C. (obtained by the distillation of 60–80°C. petroleum ether) saturated with 98% aqueous methyl alcohol. This eluant was used throughout the subsequent investigation.

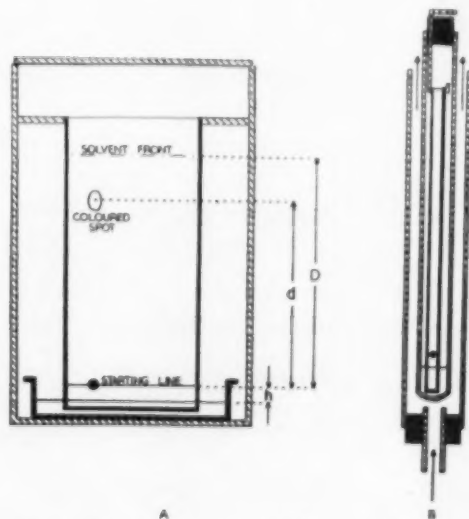


FIG. 3—Diagram of Apparatus for Chromatographic Work—
(A) Using Sheets of Paper; (B) Using Strips of Paper

2. APPARATUS

The complete arrangement is illustrated diagrammatically in Fig. 3A, which shows the paper suspended at its upper end. The lower end dips into the eluant, and the whole is housed in a rectangular vessel which was adapted from a small aquarium tank. The framework is shown separately in Fig. 4. It is of rigid construction and consists of a



FIG. 4—Elution of the Chromatogram in the Ascending Method

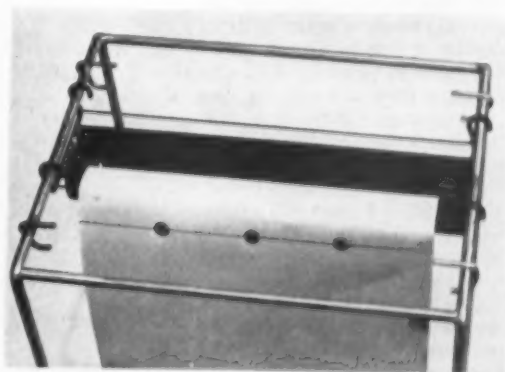


FIG. 5—Elution of the Chromatogram in the Descending Method

base plate with two troughs fixed to it. In the interior of each are two parallel rods, the purpose of which is to stabilise the paper during the elution process. Directly above the rods are the suspension clips (Kodak No. 9 hanger bars are quite suitable for this purpose). It will be seen that the paper can be suspended in two positions; the higher one is used for the saturation of the paper, and the lower position is for the elution of the

chromatogram. This arrangement ensures control over the paper during the whole process, and reduces the risk of skewing and bowing of the solvent front.

3. METHOD (ASCENDING)

A starting line is drawn with a pencil at a distance of 1.25 in. from the bottom edge of the paper. The solution of the dye in acetone is spotted on the paper with the aid of a capillary tube or a micropipette. The centre of the spot, which should be not greater than about 0.3 in. in diameter, should lie on the starting line. With a sealed tank and the paper suspended just above the liquid level, saturation is achieved in a period of 2–3 hr. When the paper, hanging freely, remains perfectly straight, this can be taken as a good indication that the saturation is complete. The paper is then lowered into the trough. The distance between the starting line and the liquid level (h in Fig. 3A) should be 0.75 in. The tank is then resealed, and left overnight, preferably at a temperature of 19–21°C. During this period of elution the solvent is soaked up by the paper, and separation of the original dye into the coloured spots or bands takes place.

Before the paper is removed from the tank the height of the solvent front is noted. After drying, a line is drawn across the paper at this height, and its distance from the starting line is measured (D in Fig. 3A). Similarly, the distance d of the centroid of the coloured spot from the starting line is found. By definition the R_f value = d/D . Although this is an inherent property of the dye, its constancy from run to run depends on a number of factors, the most important being the temperature, the eluant, and the type of paper.

A few examples of the application of the method to single components and made-up and commercial mixtures of disperse dyes are shown in Fig. 1 and 2, and the R_f values of the coloured bands are given in Table I. Fig. 1 shows chromatograms of three single components, applied one at a time and as a mixture. It will be

seen that a satisfactory resolution of the mixture into its component bands has been achieved. Fig. 2 illustrates typical chromatograms of made-up and commercial mixtures of disperse dyes.

TABLE II
Relationship between Structure and Rate of Migration on Paper of some Duranol (ICI) and Dispersol (ICI) Dyes

(The R_f values were calculated from descending paper chromatograms eluted at 19°C. for 5 hr.)

Formula	Dye	R_f Value
	Duranol Brilliant Blue CB	0.00
	Duranol Violet 2R	0.03
	Duranol Red X3B	0.04
	Duranol Red 2B	0.47
	Duranol Orange G	0.80
	Duranol Brilliant Blue G	0.84
	Duranol Blue 2G	0.92
	Duranol Red GN	0.94
	Dispersol Fast Red R	0.01
	Dispersol Fast Scarlet B	0.14
	Dispersol Fast Orange G	0.20
	Dispersol Fast Crimson B	0.24
	Dispersol Fast Orange A	0.63

All dyes used in this work were of commercial purity.

4. VARIATIONS OF THE METHOD

A number of variations of the above method have been tried and found to be successful. In each case the filter paper, the solvent, and the eluant previously described have been used, but the technique of performing the separation has been altered.

(a) Use of Paper Strips

This procedure is particularly suitable when only a single separation is to be carried out. The paper is cut into strips approx. 20 in. \times 0.5 in. The solution of a dye in acetone having been spotted on to the paper, the strip is suspended from a glass rod above the liquid, as shown in Fig. 3B, until saturated with eluant vapour. It is then lowered into the liquid and eluted overnight. The outer jacket, filled with water, helps to keep the temperature inside the inner tube reasonably constant.

(b) Descending Chromatograms

The apparatus shown in Fig. 4 can easily be adapted for descending chromatograms. By means of hooks the eluant trough is suspended from the top section of the framework (as illustrated in Fig. 5). The paper is anchored in the trough and bent over the supporting rod. The dye solution is spotted at about 3 in. from the top edge of the paper. Satisfactory chromatograms were obtained by this method in 3–5 hr. without prior saturation of the paper with eluant vapour.

(c) Use of Circular Filter Papers⁴

A circular filter paper is placed between two glass plates, the upper one having a hole cut at its centre. The plates are clamped together, and a dye solution is applied through the hole by means of a capillary tube. The eluant is delivered from a small pipette, fitted in the tripod and pressed lightly on to the paper. The rate of flow of the eluant is controlled by the absorbing power of the paper, and may be regulated, if necessary, by adjusting the pressure of the pipette against the paper to give a steady rate of migration of the coloured concentric bands.

5. USE OF THE METHOD AND DISCUSSION OF RESULTS

About 120 commercial dyes have already been examined by the method described. This number included dyes of similar structure sold under different names by various manufacturers. Dyes available in powder form were dissolved in acetone and applied to the paper as previously described. In work which was carried out on dyed fabrics, industrial alcohol (industrial methylated spirits) was used to extract the dye: in the case of nylon and Terylene boiling under reflux, and in the case of secondary cellulose acetate shaking at room temperature, were required. After evaporation of the solvent the residue was redissolved in acetone. An alcoholic solution of the dye was not suitable

for application to the paper on account of excessive spreading.

In the course of the work, it was observed that disperse dyes differed considerably in their rates of migration on paper. Firstly, we are dealing with two distinct classes of chemical compounds: the azo and the anthraquinonoid derivatives. Secondly, the nature and the position of the substituents in the aromatic nuclei have an important bearing on the rates of migration of the coloured bands and consequently on their R_f values. This point is illustrated in Table II. In the amino-anthraquinone series (Duranol dyes) free amino groups retard the rate of migration. Methyl and phenyl groups, either on the nitrogen atom or in the nucleus, considerably accelerate the rate of migration. In the azo series (Dispersol dyes) hydroxyalkyl groups slow down migration, and alkyl and phenyl groups on the nitrogen atom speed up the rate of climb.

The methods we have described have already been used successfully in testing the homogeneity of single-component disperse dyes, in determining the composition of commercial mixtures of dyes, and in studying the changes of the same dye which take place as a result of the action of light, heat, and other agencies which can cause modification of the dye. In all such cases paper chromatograms provide permanent records of each separation which are useful for reference purposes. In applying the method, we have found the eluant which we recommend to be a practical workable compromise for use with disperse dyes as a class. It is appreciated, however, that in cases where the R_f values of two or more dyes are very close together, or when a component streaks badly, it may be necessary to find an alternative eluant.

The method is being used in a number of investigations, and it is hoped to publish the results obtained in a further paper.

* * *

We wish to express our thanks to the Council and the Director of Research of the Hosiery and Allied Trades Research Association for permission to publish this paper, to the Dyestuffs Division of Messrs. Imperial Chemical Industries Ltd. for confirming the structural formulae included in Table II and supplying samples of dyes, and to Mr. P. S. Smith of the Association's staff for his assistance in the practical work.

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Equilibrium Adsorption Values of Leuco Vat Dyes on Cotton

R. H. PETERS and H. H. SUMNER

The equilibrium adsorption of a series of leuco vat dyes on cotton has been measured in various concentrations of sodium chloride and sodium hydroxide. From the results the thermodynamic affinities have been calculated. Deviations from theory are observed in so far as the affinities are not constant, but show a regular drift as the concentration of electrolyte is changed. This drift is more apparent when the added electrolyte is sodium chloride than when it is sodium hydroxide. Two explanations for these deviations from theory are proposed and discussed—either the theory is inadequate to account for the facts, or the leuco solutions of the dyes are not true solutions but are in fact aggregated.

INTRODUCTION

Quantitative information on dye adsorption by fibres is primarily of value for the representation of dyeing properties in a concise manner. Such data may be presented in a graphical form which enables the properties of a range of dyes to be assessed rapidly. Thus the amount of dye adsorbed may be plotted as a function of time, giving the rate-of-dyeing curve, or as a function of temperature, indicating the temperature-range properties.

However, the presentation of data in an assimilable form is only a first step. The next is to use the data to predict dyeing behaviour under a much wider variety of conditions than those under which the data were obtained.

One property of considerable interest in dyeing is the behaviour of dyes in admixture. For this purpose rate-of-dyeing data were collected for a large number of direct dyes, for it seemed logical that dyes with similar rates of adsorption when dyed singly would be taken up at the same rates when dyed in admixture, thus giving a dyeing which builds up on tone without noticeable separation of the components. This approach to the problem of choosing compatible mixtures of dyes was studied particularly by Lemin, Vickers, and Vickerstaff¹, but it met with only limited success. Indeed, it is apparent that it is only in exceptional cases, such as disperse dyes on cellulose acetate, that rate-of-dyeing data can be employed for this purpose with confidence. This failure is not surprising, since such arguments take no cognisance of the interaction which may occur between the dyes both on the fibre and in solution. Further, it is now clear that there are two distinct aspects of the process to be considered, viz. the maximum amount of dye taken up after long periods of time, i.e. the equilibrium uptake, and the rate at which this equilibrium is attained. There is no *a priori* reason why these should be interrelated, and hence it is necessary to determine at least two physicochemical parameters to summarise these properties and their variation with dyebath conditions.

The work presented here is restricted to studies of the equilibrium achieved between leuco vat dyes and cotton. This is a difficult system to study, but one which illustrates some of the many problems which merit further investigation.

THEORETICAL

The equilibrium uptake of dye by fibre has been examined in considerable detail by many workers, e.g. Lemin and Vickerstaff² for acid dyes on wool and Peters and Vickerstaff³ for direct dyes on

cellulose. These workers have attempted to calculate the change in free energy associated with transfer of the dye from the dyebath to the fibre rather than express their results in terms of a substantivity ratio. The reason for using the free-energy change, normally called the *affinity*, is that, formally at least, this quantity depends only on temperature and is not affected by variations in concentrations of dye and electrolyte in the bath. Thus, for the purpose of describing the equilibrium properties of a given dye-fibre system by a single parameter, insofar as that is possible, the affinity is the most useful one to derive.

The affinity, or standard free-energy change $\Delta\mu^\circ$ which occurs on the adsorption of 1 mole of dye, is related to the activity of the dye on the fibre a_f and its activity in solution a_s by—

$$-\Delta\mu^\circ = RT \ln a_f - RT \ln a_s \quad (i)$$

where R is the gas constant and T the absolute temperature.

For the purposes of computing these activities Peters and Vickerstaff divide the system into two phases, viz. the cellulose with a volume of V litres per kilogram of fibre, and the dyebath or solution phase. This device enables the amount of dye on the fibre to be translated from moles of dye per kilogram of fibre to moles of dye per litre of cellulose phase, so that all quantities are in consistent units (moles/litre). The activity terms may now be represented as products of concentrations and activity coefficients, e.g.—

$$a_f = [\text{Na}^+]_f^z \times [\text{D}^{z-}]_f \times f_f^{z+1} \quad (ii)$$

where z is the valency of the dye anion D^{z-} . The activity term is usually dropped in such calculations, since it occurs in both a_f and a_s and will to a certain extent cancel out. This argument is justified if the electrolyte concentrations in the two phases are of a similar magnitude. The terms a_f and a_s are therefore replaced by $[\text{Na}^+]_f^z [\text{D}^{z-}]_f$ and $[\text{Na}^+]_s^z [\text{D}^{z-}]_s$.

In the presence of added electrolyte the magnitude of $[\text{Na}^+]_f$ will be governed by a Donnan distribution of ions which is assumed to exist between the cellulose and the solution phases, so that when the added salt is sodium chloride this may be expressed by—

$$[\text{Na}^+]_f [\text{Cl}^-]_f = [\text{Na}^+]_s [\text{Cl}^-]_s \quad (iii)$$

where $[\text{Cl}^-]_f$ and $[\text{Cl}^-]_s$ are the concentrations of chloride ions in the fibre phase and in the dyebath respectively. This equation coupled with that for electrical neutrality in the fibre phase—

$$[\text{Na}^+]_f = [\text{Cl}^-]_f + z[\text{D}^{z-}]_f \quad (iv)$$

enables $[\text{Na}^+]_f$ to be found.

This simple picture of the system was found to be

reasonably satisfactory by Peters and Vickerstaff, who applied it to the adsorption of many direct dyes by cellulose. It is certainly as good as the treatment derived by Standing *et al.*⁴ and by Crank⁵, and it has the advantage of simplicity.

This approach was applied by Fowler, Michie, and Vickerstaff⁶ to the adsorption of a selected number of vat dyes on mercerised cotton, when they found that, by using a value of the volume term V of 0.25 litre/kg., straight lines of unit slope, as predicted by equation (i), were obtained when $\log a_t$ was plotted against $\log a_s$. In their treatment all anions in the bath other than dye anions were regarded as equivalent to chloride, and in the calculations this "equivalent chloride ion concentration" was used in equation (iii). The vat dye system was studied in more detail by Peters and Simons⁷, who carried out dyeings in the absence of sodium hydrosulphite, the dye being reduced catalytically. These authors used dye-baths containing varying quantities of added electrolyte, either sodium hydroxide or sodium chloride, and found non-linearity in the resulting $\log a_t$ - $\log a_s$ plots. In an attempt to overcome this difficulty they took into account the reduction in dye take-up due to the adsorption of the hydroxyl ions by the fibre, and by assuming that the dye and hydroxyl ions were adsorbed independently they were able to deduce the affinity of the dye. The affinity of the caustic soda is—

$$- \Delta \mu^\circ_{\text{NaOH}} = RT \ln [\text{Na}^+]_f [\text{OH}^-]_f - RT \ln [\text{Na}^+]_s [\text{OH}^-]_s \quad (\text{v})$$

This, together with the equations for the Donnan equilibrium and electrical neutrality (iii and iv), allowed $[\text{Na}^+]_f$ to be found and hence the affinity. Analysis of their results (as indicated above) for the adsorption of two dyes led to $\log a_t$ - $\log a_s$ graphs which were straight lines of unit slope. Allowance for the adsorption of hydroxyl ions is therefore essential if a reasonable value of the affinity is to be derived.

Nevertheless, in spite of this work it was thought worthwhile investigating further the adsorption of vat dyes by cellulose. Three points may be observed in the work already described. Fowler, Michie, and Vickerstaff confined their attention to a limited range of compounds (five in all) and their results were derived from experiments in which the dye concentration only was varied. No study was made of dyeing over a range of electrolyte concentrations and a range of temperatures. The work of Peters and Simons was limited to two dyes of low affinity under idealised conditions; e.g. reduction was carried out catalytically to avoid the use of sodium hydrosulphite. Furthermore, in both investigations pure dyes were used. It was therefore felt desirable to extend the data on vat dyes to include a larger range of dyes under conditions of varying electrolyte and temperature, and to use commercial samples of dyes.

EXPERIMENTAL

Materials

The dyes used in this investigation were all commercial paste-fine brands. The cellulose used was 2/20s bleached cotton yarn, which was first

seoured in a solution of soap (3 g./litre) and ammonia (sp.gr. 0.88; 1 ml./litre) at 60°C. for 20 min.

Dyeing Method

The dyes were vatted in sufficient liquor to contain all the reagents to be used in the dyeing. The vating volume varied between 25 and 40 ml. according to the amounts of sodium hydroxide, hydrosulphite, and chloride required, since these were added as stock solutions. After vating in a 4-oz. screw-capped bottle at the temperature to be used in the subsequent dyeing, the bath was made up to a standard volume of 75 ml. The yarn (0.1 g.) was then introduced, and the dyeing was carried out for a time sufficient to enable equilibrium to be reached (i.e. until test dyeings for longer periods showed no increase in dye adsorption). The times required ranged from 5 to 24 hr. according to the dye. The preparation of the dyebath and that of the dyeings were carried out under nitrogen to avoid air oxidation of the leuco dye solution.

After being dyed, the material was removed from the dyebath and squeezed to remove as much entrained liquid as possible. After being rinsed three times in a solution of sodium hydroxide (1 g./litre), sodium hydrosulphite (1 g./litre), and sodium chloride (10 g./litre) to remove any dye adhering to the surface, the dyeings were air-oxidised, rinsed in water, and then dried in an atmosphere at 65% R.H. for approx. 20 hr. before being weighed for the estimation of the dye.

The quantity of dye adsorbed was estimated by stripping the dye from a known weight of dyed fibre at 40°C. with an aqueous solution of 50% Cellosolve containing sodium hydroxide (7.2 g. per litre) and sodium hydrosulphite (10 g./litre). The dyebaths were diluted with the same mixture, and the amounts of dye in the various solutions determined optically by means of the Unicam S.P. 600 spectrophotometer.

Selected experiments representative of the various dyeing conditions were monitored by paper chromatography to ensure that no decomposition of the dye had occurred during dyeing, it being assumed that the decomposition products were coloured.

The results are given in an appendix, a copy of which may be consulted at the Society's offices.

RESULTS AND DISCUSSION

From the experimental results the ionic product $[\text{Na}^+]_f [\text{D}^-]_f$ was determined in the manner described earlier⁷, a volume term V of 0.22 litre/kg. being employed in the calculations. As is usual in this kind of work, the logarithm of the ionic product in the fibre was plotted against the logarithm of that in the bath. In all cases satisfactory straight lines were obtained, e.g. those given in Fig. 1 for Caledon Gold Orange GN at 40°C. Theory predicts that the slope of this plot should be unity, and this has been taken by other authors^{3,6} to be a criterion of agreement with experiment. In the work discussed here, the slopes were found to be, for the most part, less than unity. The actual

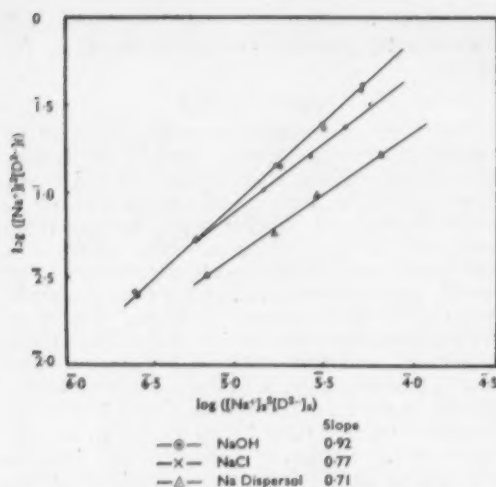


FIG. 1—Logarithmic Plots of Ionic Product for Caledon Gold Orange GN in presence of Added Electrolyte at 40°C.

values obtained, however, varied with the electrolyte added and the temperature of dyeing. Thus sodium hydroxide gave slopes very near to the theoretical, whilst all slopes obtained with sodium chloride in the dyebath were around a value of 0.8. Increases in temperature also increased the slope for most dyes. The results for a number of dyes are quoted in Tables I and II.

The affinity has been calculated for a series of dyes over the range of salt concentrations employed. There is a decrease of 3–10% in the magnitude of the affinity as the salt concentration increases (Table III). This variation is a reflection of deviations from unity in the slope of the logarithmic plots.

Slopes less than unity have been observed for some of the direct cotton dyes⁸ with a corresponding drift in the value of the affinity. The results of Geake⁹, who carried out a detailed study of the uptake of Caledon Red BN by cotton under a range of dye and electrolyte concentrations, have also been analysed by the methods described above and have been found to fall into the same pattern, i.e. a drift of the affinity with increasing concentration of electrolyte and dye. Some of these results are given in Table IV.

The drift observed in the calculated values of the affinity or the divergence of the slopes from unity is suggestive, not of experimental error, but of inadequacies of the theory. It does not seem that the use of commercial dyes is the cause, since experiments with pure dyes give similar results, as shown by the variations occurring in the values derived from Geake's experimental data.

The nature of the correction made for the adsorption of the hydroxyl ions may be questioned, since none was made by the earlier workers in this field. Calculations have been made from the data for Caledon Jade Green XN at 40°C. without any

TABLE I
Slope of Logarithmic Plot of Ionic Product in presence of Added Electrolyte at 40°C.

Caledon Dye (Paste Fine)	NaCl	NaOH	NaCl + Na Dispersol (10 ml./litre)
Red 5G ...	0.80	0.98	0.81
Yellow 4G ...	0.73	0.94	—
Red BN ...	0.84	0.92	0.78
Blue XRC ...	0.83	1.10	—
Gold Orange GN ...	0.77	0.92	0.71
Jade Green XN ...	0.80	0.91	0.59
Brilliant Purple 4R ...	0.79	1.00	—
Yellow GN ...	0.80	0.99	—

TABLE II
Slope of Logarithmic Plot of Ionic Product in presence of Sodium Chloride at Various Temperatures

Caledon Dye (Paste Fine)	25°C.	30°C.	40°C.	50°C.	60°C.	70°C.	80°C.
Red 5G ...	0.84	—	0.80	0.80	0.92	—	—
Yellow 4G ...	—	—	0.73	0.73	0.79	—	—
Red BN ...	0.80	—	0.84	0.80	0.86	0.90	0.89
Blue XRC ...	—	0.77	0.83	0.91	—	—	—
Gold Orange GN ...	—	0.79	0.78	0.87	0.83	0.82	0.80
Jade Green XN ...	—	0.77	0.80	0.81	0.84	0.86	1.00
Brilliant Purple 4R ...	—	—	0.79	0.69	0.83	—	—
Yellow GN ...	—	—	0.80	—	0.88	—	0.95

TABLE III
Effect of Salt Concentration on Affinity (kcal./mole) of Leuco Vat Dyes
(+ 0.1 M-NaOH + 0.023 M-Na₂S₂O₄ at 40°C.)

Caledon Dye (Paste Fine)	0	0.086 M.	0.171 M.	0.256 M.	0.342 M.
Red 5G ...	3.87	3.71	3.69	3.59	—
Yellow 4G ...	6.85	6.53	6.35	—	6.05
Red BN ...	4.85	4.70	4.67	4.62	4.60
Blue XRC ...	5.43	5.37	5.29	—	5.16
Gold Orange GN ...	5.42	5.24	5.07	—	5.03
Jade Green XN ...	6.17	6.11	5.99	5.90	5.91
Brilliant Purple 4R ...	6.54	6.35	6.29	—	6.32
Yellow GN ...	5.42	5.24	5.07	—	5.03

TABLE IV
Calculations based on Geake's Results⁹ on Caledon Red BN at 25°C.

Initial concn. of dye in bath $[D]_0 = 1.33 \times 10^{-4}$ M. (+ 0.05 M-NaOH + 0.01 M-Na ₂ S ₂ O ₄)		
$[Na_2SO_4]_0$ (M.)	$[D]_f$ (M.)	$-\Delta\mu^\circ$ (kcal./mole)
0	0.0812	5.38
0.1	0.147	4.96
0.2	0.188	4.86
0.3	0.229	4.85
0.4	0.264	4.86
0.5	0.284	4.83

(+ 0.05 M-NaOH + 0.125 M-Na ₂ SO ₄ + 0.01 M-Na ₂ S ₂ O ₄)		
$[D]_0$ (M.)	$[D]_f$ (M.)	$-\Delta\mu^\circ$ (kcal./mole)
1.33×10^{-5}	0.0448	5.27
2.67×10^{-5}	0.0715	5.20
6.67×10^{-5}	0.114	5.04
1.33×10^{-4}	0.160	4.93
2.67×10^{-4}	0.212	4.81
6.67×10^{-4}	0.302	4.67

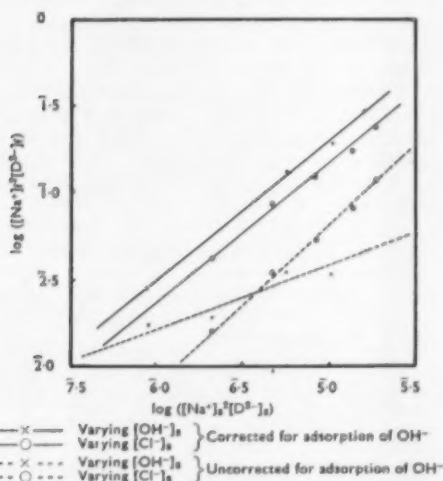


FIG. 2.—Effect of Correction for Adsorption of Hydroxyl Ions on Logarithmic Plots of Ionic Product for Caledon Jade Green XN at 40°C.

allowance for hydroxyl ions, and a slope of 0.93 has been obtained for those experiments in which the sodium chloride concentration was varied. This may be compared with the value of 0.73 obtained after making allowance for the hydroxyl ions (Fig. 2). In Fig. 2 are plotted also the data obtained when sodium hydroxide was the added electrolyte, a slope of 0.33 being obtained when no correction is made. These results, however, after allowance for the hydroxyl adsorption, fall on parallel lines which differ only slightly in position, in agreement with the conclusions of Peters and Simons⁷, and indicate the necessity of employing this correction in order that the affinity shall be regarded as a parameter characteristic of the dye-fibre system and independent of dyebath conditions.

Further, it seems unlikely that the value for the affinity of the hydroxyl ion is much in error, and the value used in this work is that determined experimentally by Peters and Simons. Indeed,

when the ratio of the hydroxyl ion in the bath to the fibre varied by a factor of 4, no change was observed in the slopes for the experiments in which sodium chloride was varied. Again, variations in the volume term from 0.22 to 0.60 litre/kg. have no apparent effect on the slope.

The explanation of the observed behaviour may lie in the solution phase of the system. It has been suggested in other work^{6,7} that aggregation of the dye in solution may occur. In the present work the slopes approached unity as the temperature was raised (Table II), an effect which could arise from disaggregation of the dyes. If this is true, increases in caustic soda concentration tend to break down aggregates, whereas additions of sodium Dispersol, giving slopes (Table I) which are farther from unity, tend to form aggregates. This last fact is not unexpected, since diffusion measurements by Valkó¹⁰ of leuco vat dye solutions in the presence of restraining agents showed the presence of large aggregates. In view of this, it is pertinent to ask whether aggregation could be a satisfactory explanation.

To investigate this point, a crude analysis has been adopted in which it is assumed that the dye solution contains concentrations of monomer $[D_1]$ and dimer $[D_2]$ in equilibrium—



The total concentration of dye in the bath $[D]_0$, used in the earlier calculations, is given by—

$$[D]_0 = [D_1] + 2[D_2]$$

The mass-law constant K is given by—

$$K = \frac{\text{Activity of dimer}}{(\text{Activity of monomer})^2}$$

which may be written as—

$$K = \frac{f_2[D_2]}{f_1^2[D_1]^2}$$

where f_1 and f_2 are the activity coefficients of the monomer and the dimer respectively. It is also assumed that the ions obey the Debye-Hückel limiting law, i.e. that the changes brought about by salt additions are governed by simple electrostatic forces. According to this law—

$$-\log f = Az^2\sqrt{I}$$

where A is a constant for all ions of valency z and I is the ionic strength. It is not possible to cancel these activity coefficients, since they occur as the first and second powers in the equation.

If an arbitrary value of K be chosen, then it is possible to calculate back to the quantities of monomer and dimer in solution. Assuming also that on account of its size and high charge the dimer is not adsorbed appreciably by the fibre, we may re-apply the simple Donnan treatment used in this work. This has been carried through for some results on Caledon Red BN using a value of K of 20 litres/mole. The large negative drift of the uncorrected results has now become a very small positive one, as may be seen from Table V.

Finally, from the variation of the affinity with temperature the heat of dyeing ΔH may be found. Examples of plots of $\Delta\mu^\circ/T$ against $1/T$ are given in Fig. 3, where it can be seen that straight lines result, the slope of these being ΔH . Some results are quoted in Table VI.

TABLE V
Affinity Values (kcal./mole) of Leuco Caledon
Red BN corrected for Aggregation

Concentration of Dye in Bath [D] _s	Uncorrected		Corrected	
	0.07 M.	0.320 M.	0.07 M.	0.320 M.
1.33×10^{-6}	—	5.27	—	5.39
2.67×10^{-6}	5.47	5.20	5.47	5.39
6.67×10^{-6}	5.39	5.04	5.41	5.36
13.33×10^{-6}	5.37	4.93	5.42	5.40
26.66×10^{-6}	5.37	4.81	5.42	5.43
66.67×10^{-6}	5.22	4.67	5.38	5.50
133.3×10^{-6}	5.17	—	5.43	—

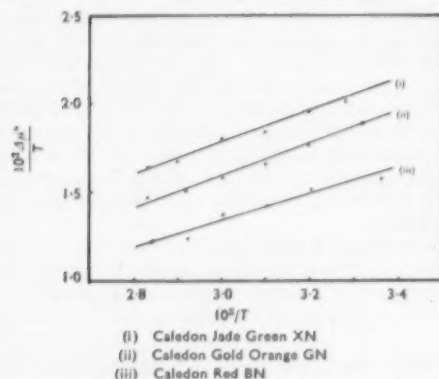


FIG. 3—Variation of the Affinity of Leuco Vat Dyes with Temperature

TABLE VI
Heats of Dyeing of Leuco Vat Dyes

Caledon Dye (Paste Fine)	— ΔH (kcal./mole)
Red BN ...	7.6
Red G ...	7.8
Jade Green XN ...	8.3
Gold Orange GN ...	9.5

CONCLUSIONS

The work described here is a further investigation of the equilibrium adsorption of leuco vat dyes by cotton. The data have been interpreted in terms of thermodynamics, and the affinities of eight dyes determined from measurements of dye adsorption in which sodium chloride and sodium hydroxide concentrations were varied. Analysis

of the data confirms that a correction for the adsorption of sodium hydroxide must be made if results of experiments in which the concentrations of both these electrolytes vary are considered. In experiments in which the concentration of sodium chloride is varied, a regular drift of the calculated values of the affinity with concentration of electrolyte occurs. This drift is less apparent when sodium hydroxide variations are considered.

Two explanations of these deviations from theory have been proposed: either the theory is inadequate to account for the facts or the solutions of dyes are not monodisperse but are in fact aggregated. Regular drifts of this nature might well be expected from the approximate nature of the theory, in which the heterogeneous fibre is replaced by a homogeneous volume phase and the ionic products of the dyes are used instead of true activities. No allowance can be made for this. However, if it is assumed that the dyes are dimerised in solution, it is possible to deduce the concentrations of each form in solution for a hypothetical value of the equilibrium constant of the dimerisation. The affinities may then be calculated on the further assumption that only the molecularly dispersed form is adsorbed. The affinities calculated on this basis are then consistent. Although this is no proof that aggregation is interfering with the application of thermodynamics, the evidence shows that the properties of dye solutions merit further study.

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(Received 19th September 1955)

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Some Observations on the Absorption of Alkali by Wool Keratin from Aqueous Solutions of Sodium Carbonate and Bicarbonate

R. P. HARKER

Sorption of alkali by wool from aqueous solutions of sodium carbonate and bicarbonate has been examined by measurement of hydrogen-ion concentrations. The effects of concentration of alkaline salt, time of immersion, liquor ratio, and temperature of equilibration have been studied in relation to subsequent measurement of the pH of an aqueous extract of a treated sample. A qualitative interpretation of results is given, and a quantitative treatment employing the Donnan theory of membrane equilibria has been attempted.

INTRODUCTION

The combination of simple acids and bases with fibrous proteins, such as wool, has been examined by a number of workers¹⁻⁶. Theoretical treatment of the combination of wool keratin with acids has been attempted by Steinhardt and co-workers⁴, Gilbert and Rideal⁶, and Peters and Speakman⁷. Gilbert and Rideal based their theory on the existence of specific sites, on which Langmuir-type adsorption* occurs. Peters and Speakman, however, applied the Donnan theory of membrane equilibria to what they have considered to be a two-phase system. These two theories, although invoking different physical phenomena, give somewhat similar results when applied to experimental data. More recently, Horner¹⁰ obtained titration data for sodium hydroxide and wool keratin and applied both theories to these. From his results, he concludes that the Donnan treatment gives better agreement with experiment, provided that tyrosine is assumed to react fully during the titration. His data also indicate a small excess of free carboxyl groups.

While this work is of considerable theoretical interest with regard to the chemical and physical properties of wool, it also has a direct bearing on the wet processing of wool in industry. The combination of wool with acids¹¹ is of interest to the dyer, whilst the effects of alkali are of concern to those occupied with the washing and scouring of raw wool, yarn, and fabric. The proposal by the International Wool Textile Organisation of a standard procedure¹² for the measurement of the pH of aqueous extracts of tops and yarns is significant of the interest shown by industry.

The present work is concerned with the uptake of alkali by wool from solutions of the alkaline salts which are most commonly used in processing, and was undertaken in an attempt to assess the usefulness of information obtained by measuring the pH of aqueous extracts of wool.

EXPERIMENTAL

The measurement of hydrogen-ion concentration was used to follow the absorption of alkali by wool keratin. The general technique applied was to

equilibrate a known weight of wool (usually 5 g. at standard condition of 16% absorption regain) in a solution of alkali of known concentration and measured pH. After equilibration for a standard time (usually 1 hr.), the wool was filtered off, and the pH of the equilibrium solution was measured. The wool samples were washed quickly with distilled water and then dried in a vacuum oven at 40°C., and the pH of aqueous extracts were subsequently determined strictly according to the I.W.T.O. specification¹².

Materials

The wool samples were taken from a bulk supply of dry-combed Australian 64s A Merino tops (scoured by a normal soap-soda process) which had been reduced to a finely divided state by a Blackfriars Junior rotary cutter; this gave a material which could be handled easily and separated from excess solution by filtering under vacuum. The wool was exhaustively extracted with polar and non-polar solvents, and then washed with distilled water until the wash-water had a pH of about 4.8-5.0.

All alkaline salts were of A.R. grade, and the distilled water used in all the experiments had a pH of 6.0-6.4.

Measurements of pH were made on a Cambridge laboratory meter, and the electrode assembly consisted of a dip-type calomel electrode and a "universal" glass electrode (Alkacid electrode: Doran Instrument Co. Ltd., Stroud, Gloucestershire). The assembly was calibrated in 0.05 M. potassium hydrogen phthalate solution (pH = 4.00 at $t = 15^\circ\text{C}$.) and 0.05 M. borax solution ($\text{pH}_t = 9.27 - 0.0085(t - 15)$). The glass electrode was calibrated in carbonate-free solutions of sodium hydroxide of known concentration. The observed pH was plotted against the pH calculated from the relation—

$$\text{pH} = \text{pK}_w + \log [\text{NaOH}] / \text{NaOH} \quad (i)$$

the data for the mean activity coefficient f being obtained from Harned and Owen¹³; this gave a straight-line relationship for the pH range 8-13. All pH measurements and aqueous extractions were carried out at room temperature (14-16°C.), apart from those measured in a thermostat at $20 \pm 0.1^\circ\text{C}$. Equilibration at 20°C . and 50°C . was controlled to $\pm 0.1^\circ\text{C}$. in a thermostat, the other samples being equilibrated at 14-16°C.

* As the general terminology of sorption phenomena is only loosely defined, the following terms have been adhered to in this paper. Sorption has been used to describe any transfer from solution to the "solid" phase, and desorption to describe the reverse process. Adsorption, normally implying some degree of penetration, has been employed to describe general uptake by the wool fibre. The term adsorption, often reserved for purely surface sorption, has been used only in connection with Langmuir-type sorption on specific sites.

RESULTS

Typical experimental results are represented in Fig. 1-7. The general conclusions to be drawn from these are as follows—

(i) *Effect of Variation in Liquor Ratio during Equilibration*
(Fig. 1 and 2)

It is clear that, for sodium carbonate systems, the pH of aqueous extracts at lower liquor : wool ratios is higher than the equilibrium pH, but that the reverse is true at higher ratios. The pH of aqueous extracts does not appear to vary a great deal over a wide range of liquor ratios.

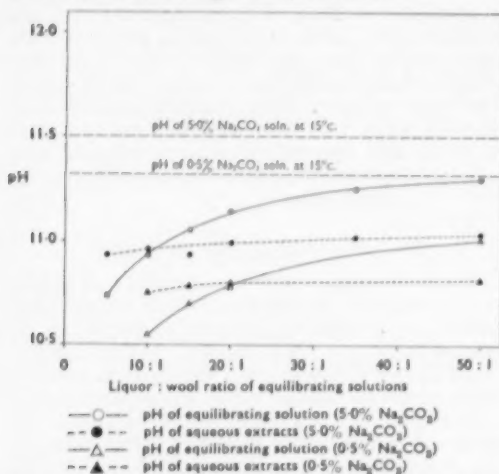


FIG. 1—Effect of Liquor:Wool Ratio of Equilibrating Solutions on pH of Aqueous Extract

(ii) *Effect of Temperature of Equilibration*
(Fig. 2)

For sodium carbonate systems, the pH of the aqueous extract for a particular liquor ratio is lower for wool equilibrated at higher temperatures, and the temperature effect is more marked at low liquor ratios.

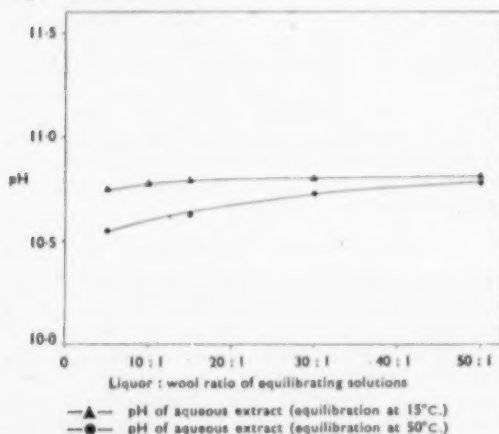


FIG. 2—Effect of Temperature of Equilibrating Solutions (0.5% Na_2CO_3) on pH of Aqueous Extracts

(iii) *Effect of Concentration of Equilibrating Solution*

(Fig. 3 and 4)

With sodium carbonate solutions, the pH of the aqueous extract may be higher or lower than the equilibrium pH (Fig. 1), but the graph of pH of the aqueous extract against concentration follows the same type of curve as the plot of equilibrium pH against concentration. These will probably coincide under a given set of conditions (Fig. 3).

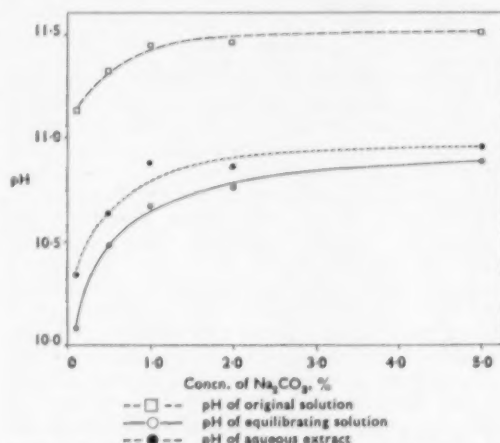


FIG. 3—Effect of Concentration of Equilibrating Solution on pH of Aqueous Extract (Liquor ratio = 15:1; temperature = 50°C.)

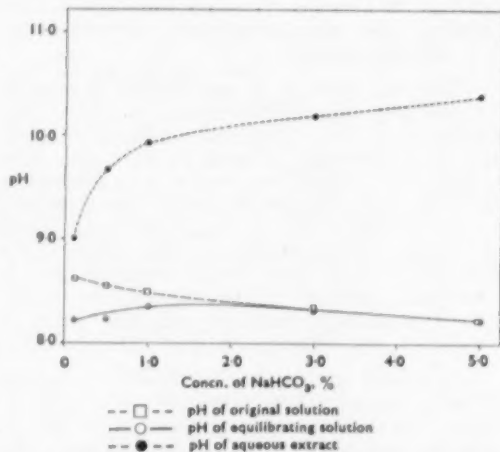


FIG. 4—Effect of Concentration of Equilibrating Solution on pH of Aqueous Extract (Liquor ratio = 15:1; temperature = 20°C.)

For sodium bicarbonate, however, the pH of the aqueous extract is always much higher than the equilibrium pH, especially at high concentrations (Fig. 4).

(iv) *Effect of Time of Equilibration*
(Fig. 5)

The uptake of alkali seems to be very rapid (Fig. 5), the greater part being taken up during the

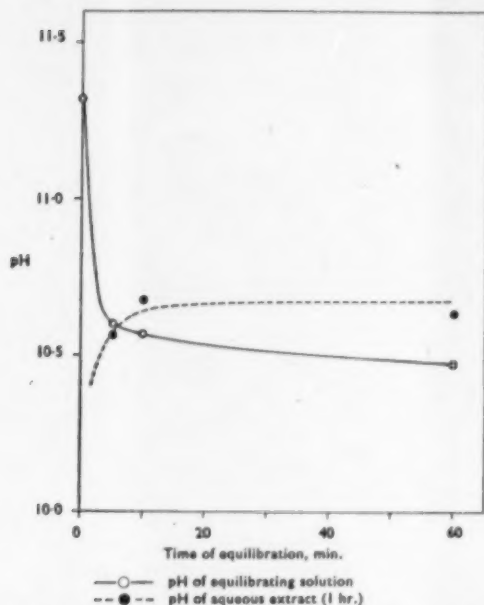


FIG. 5.—Effect of Time of Equilibration on pH of Aqueous Extract (0.5% Na_2CO_3 ; liquor ratio = 15:1; 50°C.)

first 10 min. This is borne out by the pH of the corresponding aqueous extracts.

(v) *Effect of Time and Number of Extractions*
(Fig. 6 and 7)

Desorption of alkali appears to be a rather slower process than its sorption (Fig. 6). Repeated extraction of samples equilibrated in a series of

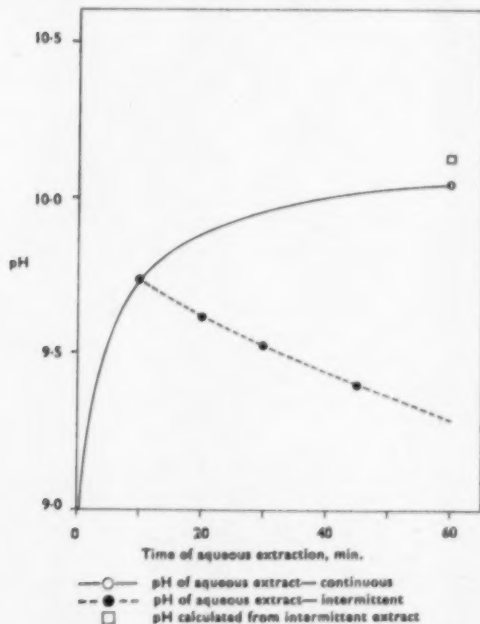


FIG. 6.—Effect of Time of Aqueous Extraction on pH (0.725% KHCO_3 ; liquor ratio 50:1; 15°C.)

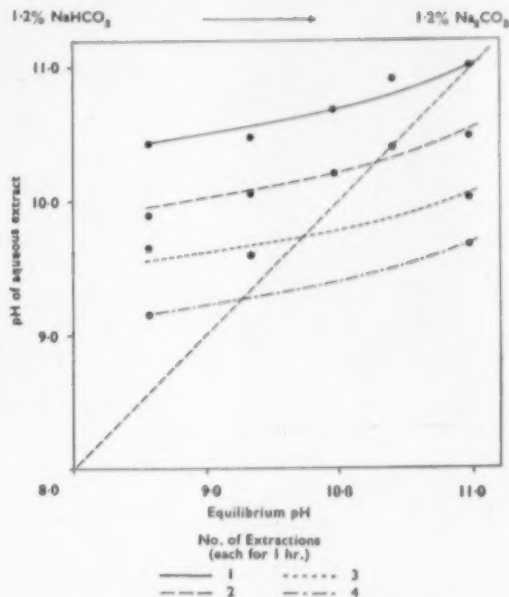


FIG. 7.—pH of Aqueous Extracts of Wool equilibrated in Sodium Bicarbonate-Sodium Carbonate Buffer Mixtures

sodium carbonate-sodium bicarbonate buffer mixtures gave a number of curves of the same general form, the pH of the aqueous extract decreasing along the series as the number of extractions increased (Fig. 7).

(vi) *Effect of Soap*
(Table I) (p. 20)

Reference to Table I shows that the pH of an aqueous extract of wool equilibrated in soap solution is due entirely to the uptake of alkali supplied by the soap. In the presence of alkali, the soap exerts very little effect. Soxhlet extraction with benzene-methanol solvent (azeotropic mixture—3 vol. of benzene:2 vol. of methanol, b.p. 58.3°C.) does not remove any appreciable amount of absorbed alkali.

DISCUSSION

Consideration of the reactions which may occur when wool is introduced into an aqueous solution of an alkali salt, such as sodium carbonate, is necessary in order to interpret the above results. It is an established fact that wool will absorb hydroxyl ions, and sodium ions will be taken up by the fibre to preserve electrical neutrality. This can be represented as a net uptake of sodium hydroxide—



Thus, the final result of introducing wool into sodium carbonate solution is the absorption of sodium hydroxide and the appearance of an equivalent amount of sodium bicarbonate. This will be accompanied by a significant drop in the pH of the sodium carbonate solution at equilibrium, which will be more apparent at low salt concentrations and/or low liquor ratios. The fall in pH

TABLE I

Treatment of Wool Samples	Original Solution (a)	pH Values Equilibrium Solution (b)	Aqueous Extract (c)	Benzene-Methanol Extract (% on wt. of Wool) (d)
(1) Equilibrated for 1 hr. in 0.5% Na ₂ CO ₃ soln. (at 15°C. and 15:1 liquor ratio)	11.32	10.61	10.84	—
(2) Sample as for (1) extracted for 3 hr. with benzene-methanol	11.32	10.61	10.83	Blank figure
(3) Equilibrated for 1 hr. with 0.05 M Na ₂ CO ₃ + 0.05 M-KCNS (at 15°C. and 15:1)	—	10.57	10.72	—
(4) Equilibrated for 1 hr. with 0.1% Na stearate (tech. pure) (at 15°C. and 15:1)	10.70	9.58	9.38	—
(5) As for (4), extracted for 3 hr. with benzene-methanol	10.70	9.58	9.32	Soap 0.057% Free fatty acid 0.119%
(6) Equilibrated as (1). Then soln. from 1(b) made up to 0.1% Na stearate and equilibrated for a further 1 hr.	—	10.47	10.83	—
(7) As for (6), extracted with benzene-methanol for 3 hr.	—	—	10.67	Soap 1.313% Free fatty acid 0.215%
(8) Equilibrated for 15 hr. with 0.1% pure Na myristate (at 20°C.)	9.79	9.05	8.60	—

in such cases can be calculated from the relation—

$$\text{pH} = \text{p}K_a + \log \left(\frac{[\text{Salt}] - [\text{OH}]}{[\text{Acid}] + [\text{OH}]} \right) \text{ for } \text{pH} > 10.0 \quad (\text{ii})$$

where [OH] is the concentration (or activity) of hydroxyl ions in solution. When the initial and final pH values are known and the initial concentrations of salt (Na₂CO₃) and acid (NaHCO₃) are known, we have—

$$\begin{aligned} \text{pH}_1 - \text{pH}_2 = \log \left(\frac{[\text{Salt}]_1 - [\text{OH}]_1}{[\text{Acid}]_1 + [\text{OH}]_1} \right) \\ - \log \left(\frac{[\text{Salt}]_2 - [\text{OH}]_2}{[\text{Acid}]_2 + [\text{OH}]_2} \right) \quad (\text{iii}) \end{aligned}$$

Approximate values of [OH] for various pH values can be obtained from equation (i). Alternatively, the concentration of acid present at any pH value for a definite carbonate-bicarbonate system can be determined experimentally by titration of the required salt solution with mineral acid, or by back-titrating the salt-acid mixture with sodium hydroxide until the pH value for a solution of Na₂CO₃ (at a particular concentration) is reached.

As the amount of bicarbonate produced is equivalent to the amount of base combined with the wool, the latter can be readily calculated using equation (iii) and the following expression—

$$[\text{Salt}]_1 = [\text{Salt}]_2 + [\text{Acid}]_2 \quad (\text{iv})$$

INTERPRETATION OF RESULTS

Effect of Time

The general conclusion that the sorption of alkali is a rather faster process than desorption can be attributed to concentration effects in the external (equilibrating) solution. Sodium hydroxide alone is a much simpler system¹⁰; the amount of sodium hydroxide required to produce a given pH is much less than the concentration of sodium carbonate necessary to produce the same pH in the external solution. Hence, there is a much greater salt concentration outside the fibre than when sodium hydroxide alone is

used for equilibration. In the case of desorption, where very small amounts of sodium hydroxide are being removed from the fibre, "internal" and external solutions will have small concentrations with small differences in ionic strength.

Effect of Temperature

In the case of sodium or potassium hydroxide, an increase in the temperature of equilibration is accompanied by an increase in absorption of base for a given pH value^{4,10,11}. However, with sodium carbonate results based on the pH values of aqueous extracts indicate that the reverse is true, although differences are small. The explanation of this probably lies in the variation of the pH of sodium carbonate solutions with temperature. The $d(\text{pH})/dT$ coefficient is negative for such systems, and an increase in temperature will cause a significant decrease in hydroxyl-ion concentration, with the result that, for a given solution, equilibration at elevated temperature will actually take place at a much lower pH value than equilibration at room temperature. Degradation of the wool protein (which should be very slight at these pH values) will also reduce the value of the pH of aqueous extracts.

Effect of Concentration of Sodium Carbonate

As would be expected, the pH of the aqueous extract is related to the concentration to the extent that higher values for extracts are recorded from wools equilibrated at higher concentrations. However, the liquor ratio of equilibration is usually the factor determining whether the pH of the extract is higher or lower than the equilibrium pH. This will be dealt with below.

Effect of Liquor: Wool Ratio

The apparently paradoxical state of affairs, that the pH of the aqueous extract at low liquor ratios is higher than the equilibrium pH, whilst that at high liquor ratios is lower than the equilibrium pH, demands an explanation. In this case,

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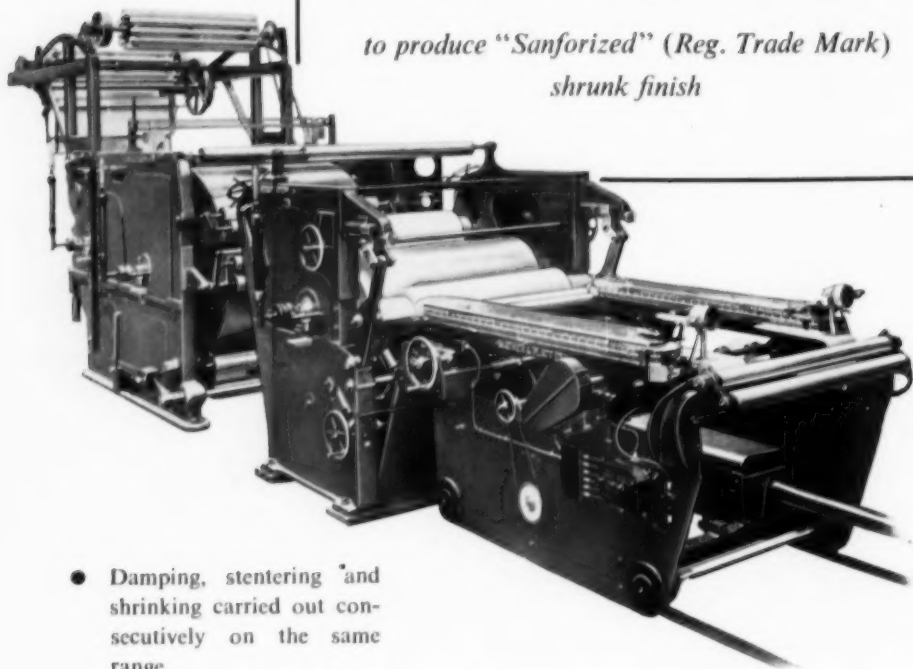
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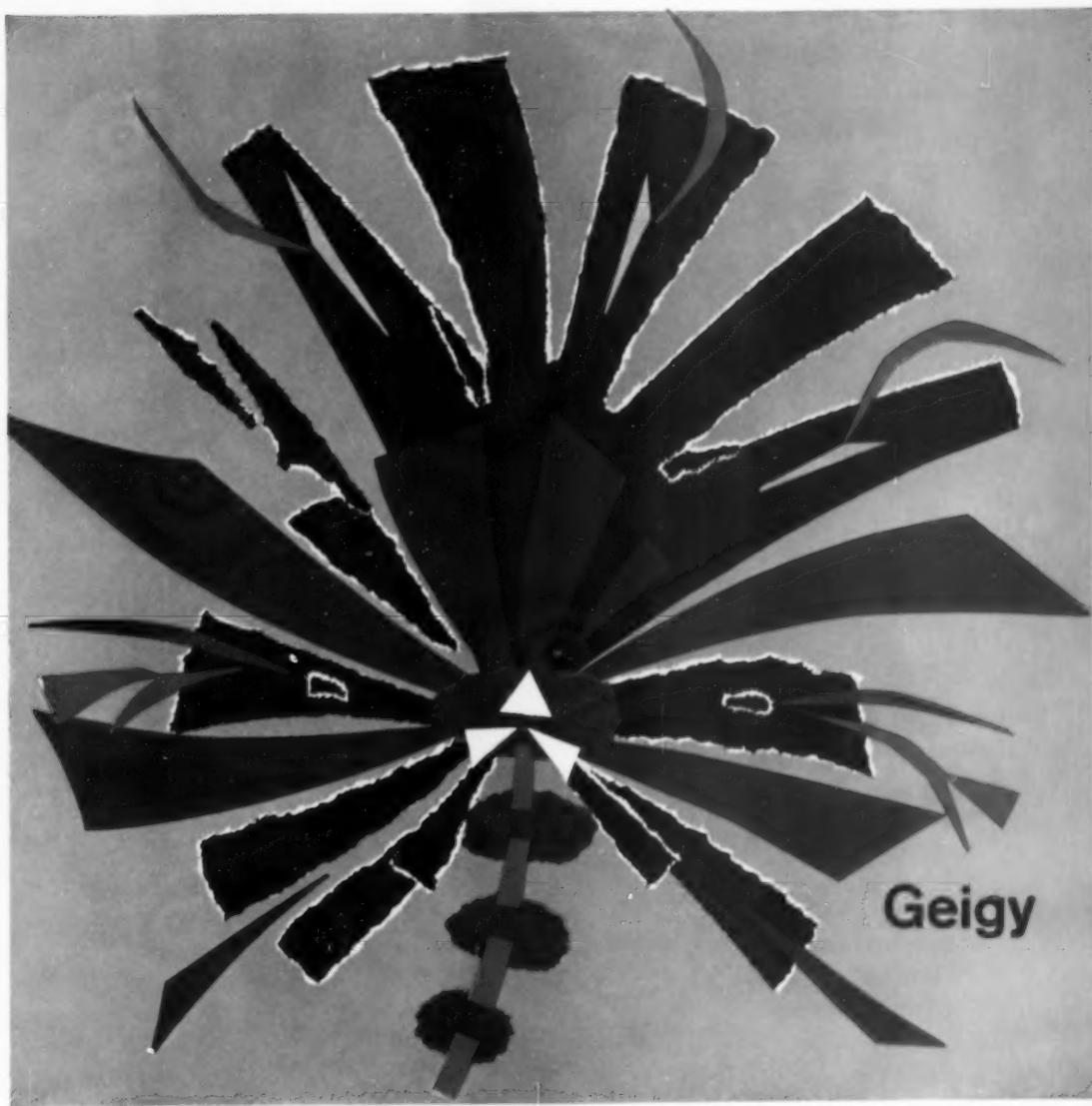
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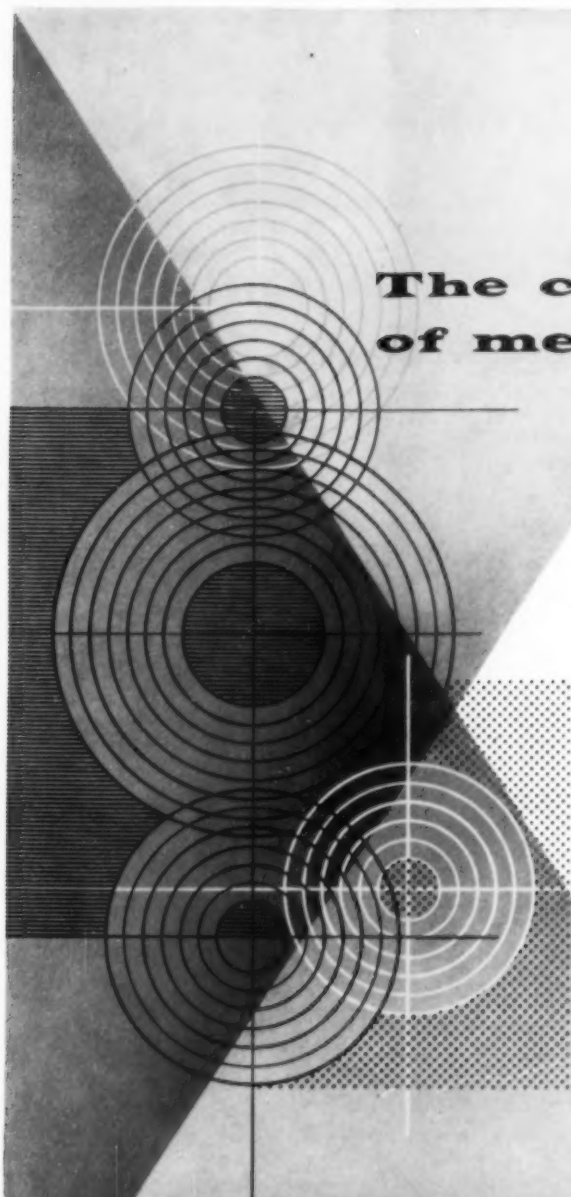
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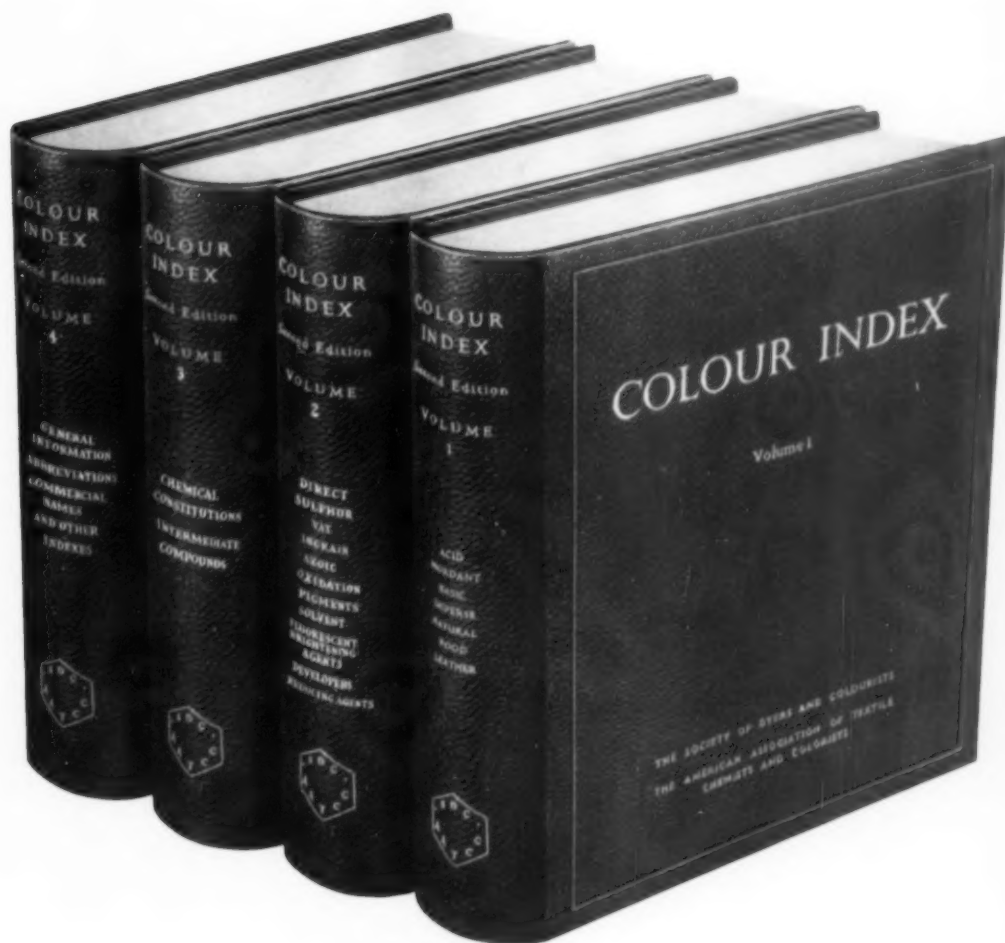
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- A—(American) The methods of The American Association of Textile Chemists and Colorists.
- B—(British) The methods of The Society of Dyers and Colourists.
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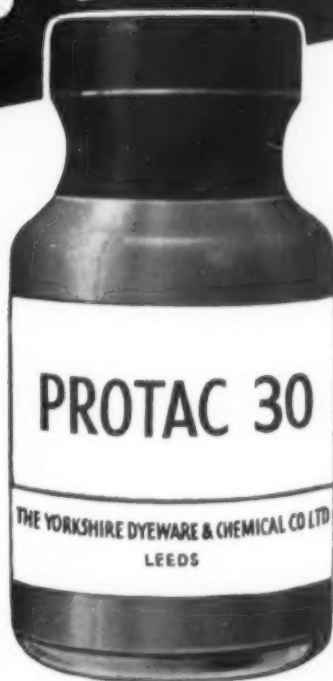
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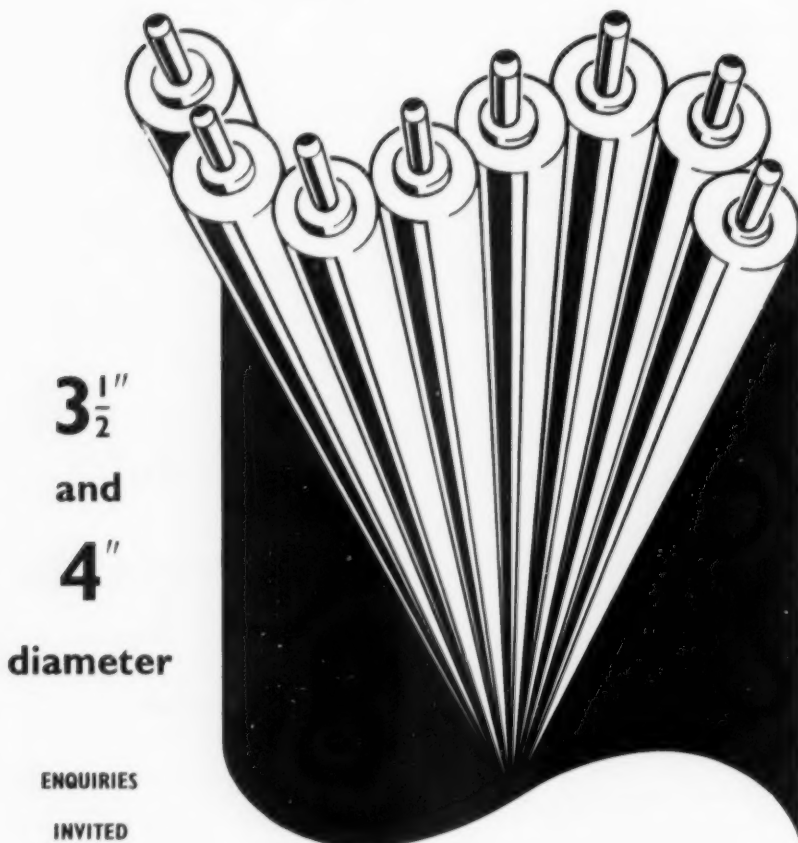


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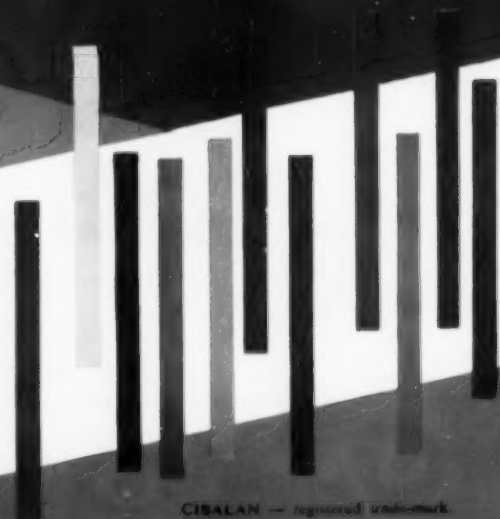
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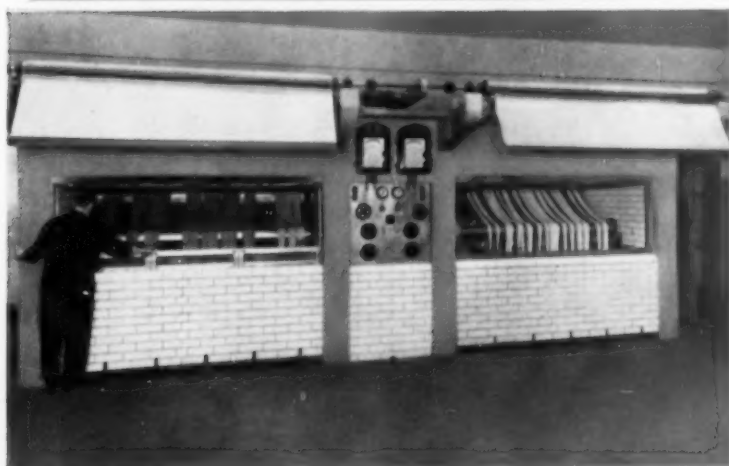


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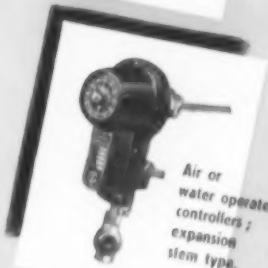
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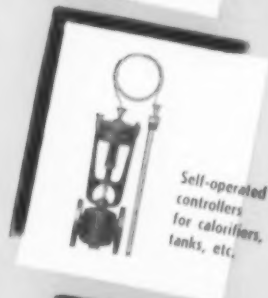
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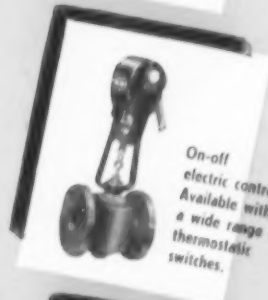
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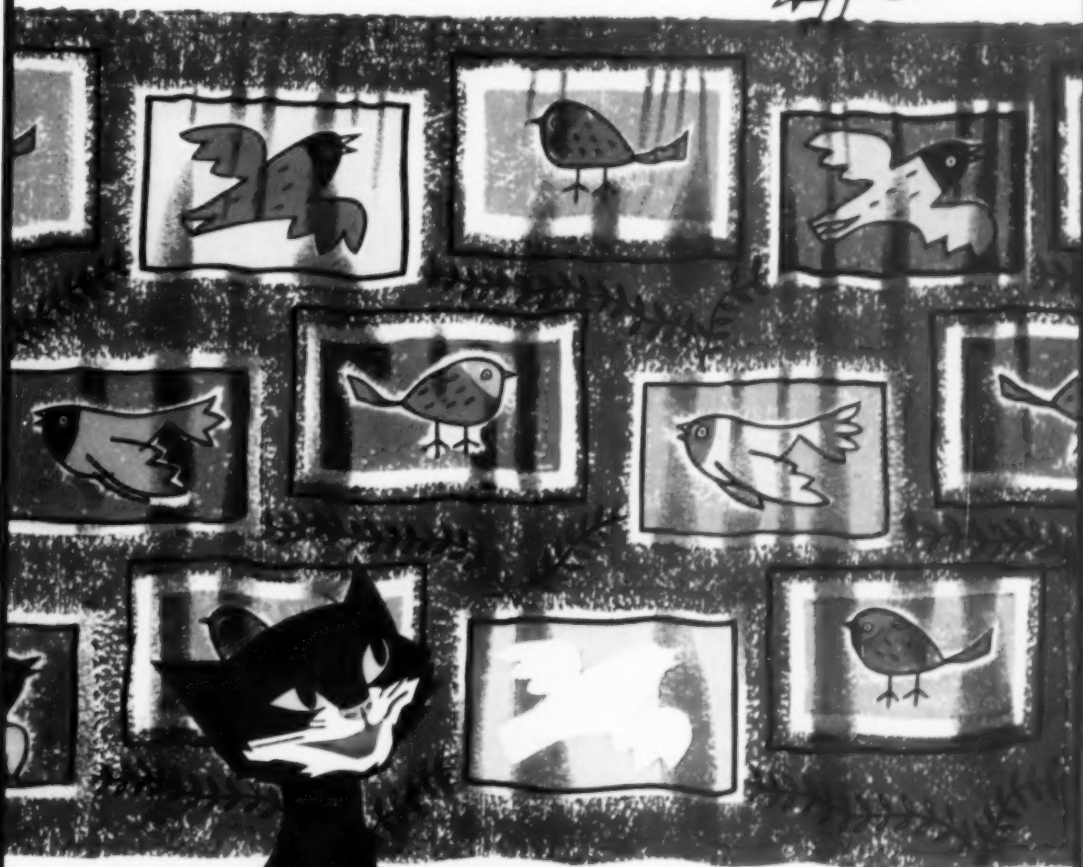
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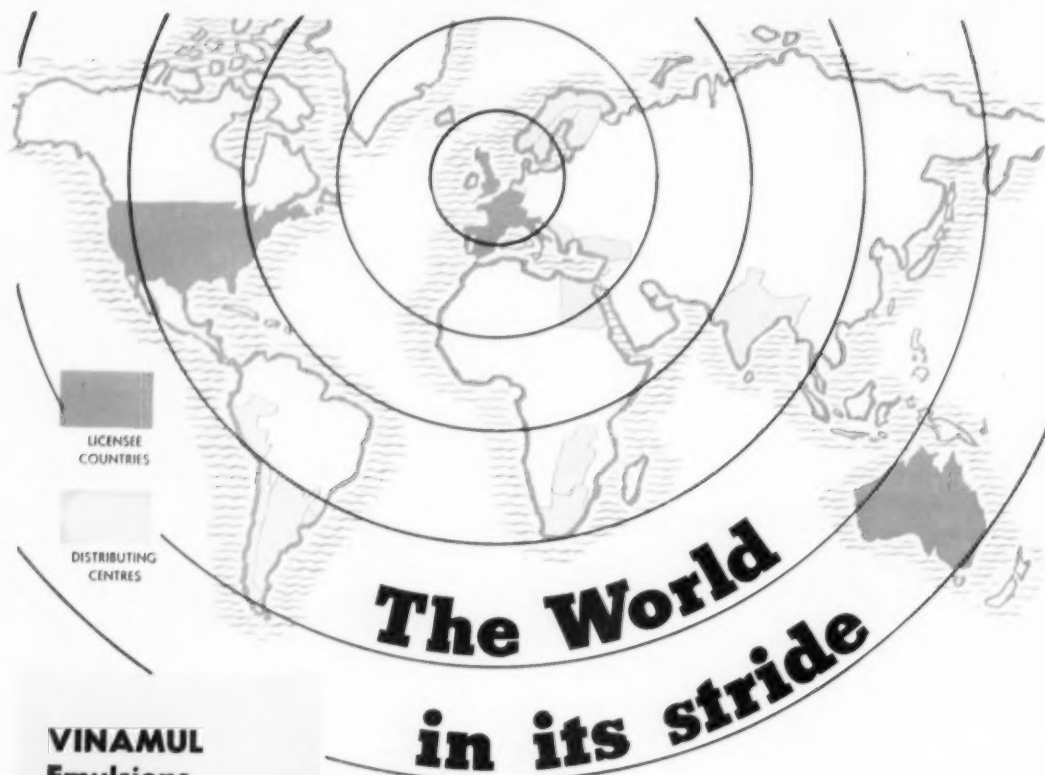
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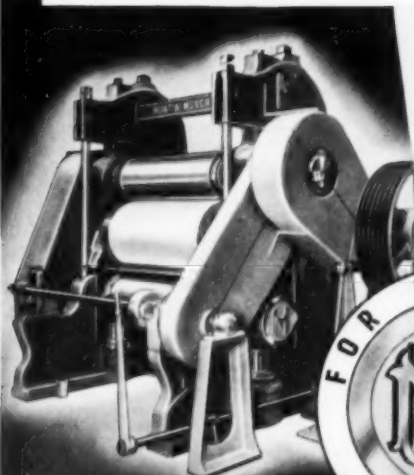
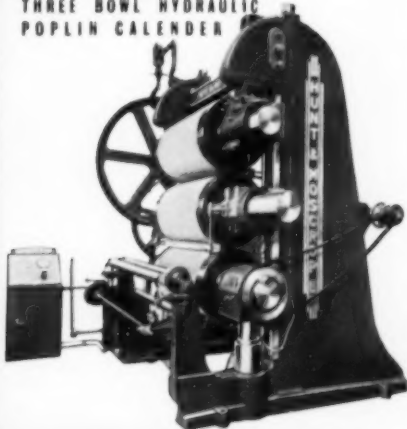


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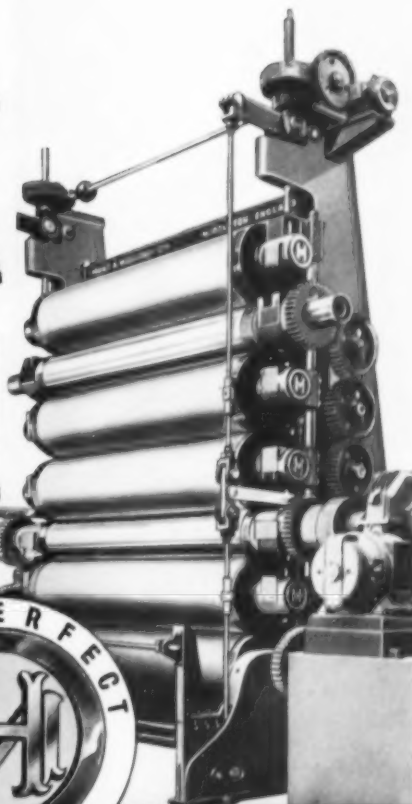
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the pH of the aqueous extract does not differ markedly over a wide range of liquor ratios, but the equilibrium pH values differ greatly. It is reasonable to assume, from the pH values of the aqueous extracts, that the amount of alkali taken up in each case is not very different. The wide variation in equilibrium pH is due to approximately the same amount of sodium bicarbonate being produced in each case, but giving a widely different carbonate-bicarbonate ratio. This follows from the earlier discussion and is proved by the results shown in Table II. As can be seen, the amount of sodium bicarbonate produced is roughly the same in each case, whilst the carbonate-bicarbonate ratio varies widely. The amount of base taken up is also very similar, and the pH of the aqueous extract agrees with this.

tion of equation (iii) is not very satisfactory. However, titration data have enabled the amounts of combined base to be determined. For example, 0.075 milliequivalent of combined base per gram of wool was found after equilibration in 1.0% sodium bicarbonate (equilibrium pH = 8.35) at 20°C. with a liquor ratio of 15 : 1.

Application of the Donnan Theory

Peters and Speakman⁷ have applied the Donnan theory to the sorption of acids by wool: their general treatment includes the cases of salt-free absorption and absorption in the presence of added salts. This has been extended by Horner¹⁰ to include the absorption of simple alkalis, with and without the addition of uniunivalent salts. In the consideration of sorption from sodium carbonate

TABLE II
Equilibration of Wool (5 g.) in Various Liquor Ratios

Ratio	Liquor Vol. (ml.)	pH _i	NaHCO ₃ produced (g.)	Na ₂ CO ₃ remaining (g.)	Alkali taken up by Wool (m-equiv. NaOH per g.)	pH of Aqueous Extract
(a) 0.5% (0.047 M.) Na ₂ CO ₃ at 50°C. pH _i = 11.32						
5 : 1	25	10.03	0.031	0.064	0.085	10.55
15 : 1	75	10.48	0.042	0.317	0.117	10.63
30 : 1	150	10.78	0.044	0.690	0.118	10.73
50 : 1	250	10.89	0.053	1.174	0.146	10.78
(b) 5% (0.472 M.) Na ₂ CO ₃ at 15°C. pH _i = 11.50						
5 : 1	25	10.73	0.090	0.713	0.247	10.93
10 : 1	50	10.93	0.108	1.963	0.298	10.96
15 : 1	75	11.05	0.109	3.212	0.300	10.93
20 : 1	100	11.14	0.104	4.643	0.287	10.99
35 : 1	175	11.25	0.107	8.212	0.295	11.02
50 : 1	250	11.30	0.116	11.960	0.319	11.03

The values in Table II also show the effect of salt concentration on the absorption of alkali. The values in Table II(a) were calculated from equations (iii) and (iv). In this case, the hydroxylion concentration was calculated from equation (i), the mean activity coefficients for sodium hydroxide being employed. In Table II(b), however, the amount of sodium bicarbonate produced was found from titration data at the requisite concentration and pH.

Absorption from Sodium Bicarbonate Solutions

In general, the sorption of alkali from solutions of sodium bicarbonate follows the same pattern as that from sodium carbonate solutions. However, the desorption curve (pH of aqueous extract) gives pH values very much in excess of the equilibrium values. In this case it is assumed that absorption of alkali (NaOH) must take place, as sodium ions are produced by dissociation of the bicarbonate. Thus, the hydrolysis of the sodium bicarbonate gives the net result—



Desorption of this alkali will give a solution of sodium hydroxide alone, at a higher pH value than the corresponding equilibrium pH of the strongly buffered bicarbonate solution.

The fall in pH from the original solution to the equilibrium solution is usually small, and applica-

solutions, the system is rather more complex, as there is virtually no case of "salt-free" absorption. Furthermore, after sorption has taken place, a new ionic species (HCO_3^-) has appeared. The state at equilibrium can be represented as—

Fibre Phase				Solution Phase	
$-\text{COO}^-$	A			$[\text{Na}^+]$	$= C$
$-\text{NH}_3^+$	$A(1 - \theta_1)$	$[\text{Na}^+]$	$= c$	$[\text{OH}^-]$	$= D$
$-\text{NH}_2$	$A\theta_1$	$[\text{OH}^-]$	$= d$	$[\text{HCO}_3^-]$	$= F$
$-\text{OH}$	$T(1 - \theta_2)$	$[\text{HCO}_3^-]$	$= f$	$[\text{CO}_3^{2-}]$	$= G$
$-\text{O}^-$	$T\theta_2$	$[\text{CO}_3^{2-}]$	$= g$		
Volume = v				Volume = V	

Here, tyrosine has been included in the combining groups and θ represents the fraction of each of the groups which have combined. C , D , F , and G are expressed in gram-equivalents per litre, and c , d , f , and g in milliequivalents per gram of dry wool; v is in millilitres per gram of dry wool.

By definition, the distribution of ionic species between fibre and solution for thermodynamic equilibrium can be obtained by equating chemical potentials¹⁴. If ψ is the potential difference between fibre and solution, the distribution coefficient λ between the electrically charged phase is—

$$\lambda = \exp \left(\frac{\psi F}{RT} \right)$$

and gives rise to the relations—

$$\lambda = \frac{C}{e} = \frac{f}{F} = \sqrt{\frac{g}{G}} = \frac{d}{D} \quad (v)$$

For the condition of electrical neutrality in the external solution—

$$F + 2G + D - C = 0 \quad (vi)$$

The amount of base combined with the fibre is usually calculated by subtracting the residual base from the amount originally present. The parameter B can be defined⁷ as—

$$\frac{B}{v} = (e - C) - (f - F) - 2(g - G) \quad (vii)$$

and can be shown to be related to the combining capacity of the wool.

Using the relations in equation (v) to eliminate the "internal concentrations" e, d, f , and g in (vii), it follows that—

$$\frac{B}{v} = \left(\frac{1}{\lambda} - 1\right)C - (\lambda - 1)F - 2(\lambda^2 - 1)G \quad (viii)$$

Combining (vi) and (viii)—

$$\frac{B}{v} + D = \frac{e}{\lambda} - F\lambda - 2G\lambda^2 \quad (ix)$$

Application of the Donnan theory assumes an "internal" solution with ion concentrations different from those of the external solution. This concept has been used by Peters to calculate internal pH values for acid absorption. Thus the relation $\lambda = d/D$ (v) can be written—

$$\lambda = \text{pOH}_{\text{ext}} - \text{pOH}_{\text{int}} \quad (x)$$

Now all the quantities in equation (ix) are known, or can be calculated, with the exception of λ . In practice, the solution of equation (ix) is best effected by graphical means. Values of C, D, F , and G for a particular value of B/v were put into the equation and curves of B/v against—

$$\frac{e}{\lambda} - F\lambda - 2G\lambda^2 - D$$

were constructed using values of λ from 0.01 to 1.0. The value of λ for the particular B/v fraction was then found from the graph. The values of λ and pOH_{ext} being known, pOH_{int} was found from equation (x), and compared with Horner's data (Fig. 8) calculated from equations (xi) and (xii), which can be obtained from equation (ix). Thus, if F and $G = 0$ and $C = D$, (ix) is reduced to the form—

$$\text{pOH}_{\text{int}} = 2\text{pOH}_{\text{ext}} + \log \left(\frac{B}{v} + D \right) \quad (xi)$$

which relates to salt-free absorption; or, in the presence of salt—

$$\text{pOH}_{\text{int}} = \text{pOH}_{\text{ext}} - \log C + \log \left(\frac{B}{v} + D \right) \quad (xii)$$

The value of v (internal volume) was taken to be 0.285 ml./g.; this has been shown⁷ to be the most likely figure for the volume of imbibed water at neutral pH.

CONCLUSION

The main conclusion that can be drawn from this work is that, although the pH of an aqueous

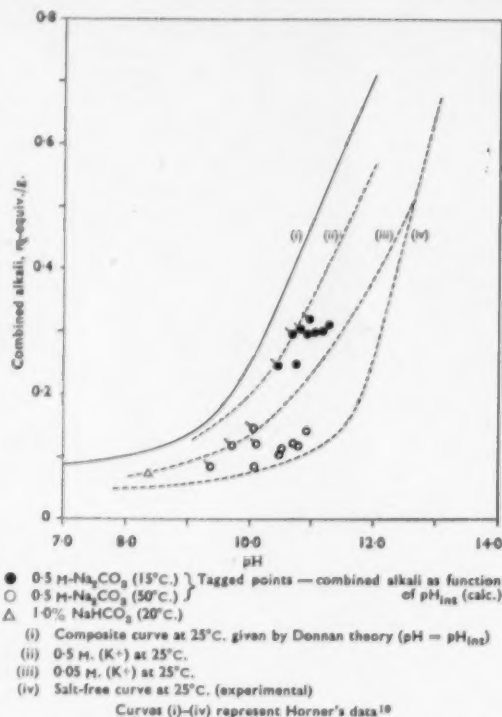


FIG. 8—Effect of Ionic Strength on Absorption of Alkali by Wool

extract of wool can be regarded as a measure of combined base, it does not provide any definite information on the previous history of the wool. Thus, depending upon liquor ratio, temperature, and time of immersion, a strong bicarbonate and a weak carbonate solution could give rise to the same pH of the aqueous extract. This observation will apply to the practical operations of washing raw wool and scouring yarn and cloth. In the case of yarns and fabrics the previous history with regard to alkali treatment will affect the system. Soap, in the presence of alkaline salts, does not appear to influence the system greatly, but it functions as a supply of alkali in their absence. Application of the Donnan theory does not give very good agreement with Horner's data for the saturation absorption of sodium hydroxide, but the $(\text{pOH}_{\text{ext}} - \text{pOH}_{\text{int}})$ shift predicted by equation (ix) is of the order to be expected. Better agreement might be obtained if sufficient data were available for activities of the various ion species under consideration. In very dilute solutions, the mean activity coefficient for sodium hydroxide¹³ was used where applicable, but the absence of reliable information for carbonate-bicarbonate systems may have introduced inaccuracies in calculations. Furthermore, although no trace of CO_3^{2-} could be detected in aqueous extracts, this does not preclude the possibility that some carbonate is absorbed.

The author is grateful to Miss S. Braithwaite for assistance with experimental work, to Mr. J. A.

Medley for valuable discussion, and to Dr. F. O. Howitt for helpful comment.

WOOL INDUSTRIES RESEARCH ASSOCIATION
"TORRIDON"
LEEDS 6

(17th May 1956)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

The Effect of Solvents in Dyeing

The increase in the rate of dyeing of wool at low temperatures, produced by adding organic solvents to an aqueous dyebath, has been attributed to disaggregation of the dye by the dissolved solvent in the aqueous solution. Solvents which are completely miscible with water, e.g. those specified by Ciba Ltd.¹, must certainly act in this way. Solvents which are only partially miscible, however, act mainly by being adsorbed as a surface phase in the fibres, the dye rapidly dissolving in this surface solvent phase and diffusing from there into the fibre more rapidly than it would from the bulk aqueous phase.

One can devise an interesting converse experiment to support this interpretation. Suppose a water-soluble dye is dispersed in a solvent in which it is insoluble and then brought into contact with a fibre, e.g. cellulose, which has little or no affinity for the solvent but considerable affinity for the dye and for water; under these conditions hardly any dyeing should take place. If water is now added to the system, it should be adsorbed by the fibre and thus provide a surface phase in which the dye can dissolve and from which it may then diffuse into the fibre. The water here would then behave in a similar manner to the organic solvent when the latter is adsorbed by wool from a water-solvent dyebath.

A hank (5 g.) of bright viscose rayon staple yarn was immersed in *n*-butyl alcohol (210 c.c.) to which had been added a concentrated solution of Chlorazol Sky Blue FFS (ICI) (12.5 c.c. of a 0.4% aqueous solution of commercial dye) and water (27.5 c.c.), the temperature being 60°C. The material was vigorously agitated in the dyebath and very rapid uptake of the dye occurred, exhaustion being virtually complete in 2 min.

The experiment was then repeated, the *n*-butyl alcohol being replaced by an equal volume of water. From this dilute aqueous dyebath the rate of uptake of dye, as assessed by the colour of the solution and the depth of the dyeing, was very much lower than in presence of *n*-butyl alcohol, a considerable amount of dye remaining in the solution after 30 min.

Since in this second experiment the amount of water present was several times greater than in the

first case, a third experiment was carried out in which the *n*-butyl alcohol was omitted but no additional water was added, dyeing being carried out in the relatively concentrated dye solution. In this instance the uptake of dye was more rapid than in the second experiment but still much less rapid than in the first case.

Further experiments using bleached, mercerised cotton cloth showed the same result, and Chlorazol Blue G 150 behaved in the same manner as Chlorazol Sky Blue FFS. When *n*-amyl alcohol, *n*-hexyl alcohol, and cyclohexanol were also used instead of *n*-butyl alcohol, similar increases in the rate of uptake of dye were observed, the maximum effect apparently coinciding with the presence in the system of an amount of water certainly not greater than that required to saturate the fibre and the solvent under the conditions used. The commercial brands of Chlorazol Sky Blue FF and Chlorazol Blue G which were used were insoluble in *n*-butanol. These are tetrasulphonated direct dyes. Commercial Chrysophenine GS, which is disulphonated, was found to be soluble in this solvent and when it was used instead of the tetrasulphonated dyes the differences in rate of uptake from solvent-water mixtures and from water only were hardly noticeable under the experimental conditions employed. The effect of adding more water to an *n*-butanol-water-cellulose system was similar to that of adding excess of *n*-butanol to a water-*n*-butanol-wool system; in both cases the rate of uptake of dye is diminished, dye remaining in the excess water or butanol, respectively, out of contact with the fibre.

L. PETERS
C. B. STEVENS

DEPARTMENT OF TEXTILE INDUSTRIES
DEPARTMENT OF COLOUR CHEMISTRY AND DYEING
THE UNIVERSITY
LEEDS 2

1st November 1956

¹ Ciba Ltd., BP 669,192.

Fading Lamps

In the November *Journal* it is stated¹ that fading lamps were "first marketed in 1919". This is not true. I used a German-made fading lamp in the dyeing laboratories of Read Holliday & Sons

Ltd. at Huddersfield in the early nineteen-hundreds. It consisted of a 6-8 in. plano-convex lens which collected the light from a naked arc lamp, which was then focused through a similar-sized double convex lens on to the pattern-holder. All three were mounted on one long graduated arm, which could be tilted and adjusted as desired. I ordered it from a description and illustration in Lehne's *Färber-Zeitung*. I presumed the German dye firms were using it at that time.

Compared with the fading lamps of today it was crude, but in my view it was the first fading lamp marketed.

C. M. WHITTAKER

1 WEAPONNESS PARK
SCARBOROUGH

25th November 1956

¹ McLaren, K., J.S.D.C., 72, 527 (Nov. 1956).

Rapid Dyeing

It is almost a certainty today that any technical journal, like our own, will contain short or long commentaries on aspects of "continuous processing". This fact brings to mind that the only appropriate shade-cards, so excellently prepared by the dyemakers, are those referring to "padding". And they are few indeed. I am not aware that the dyemakers have offered shade-cards relating to the dyeing of chain-warps or the continuous dyeing of cloth. I agree that they can hardly be expected to do so, since such forms of processing are done on a variety of machines running at varying speeds according to depth of dyeing, liquor ratio, and so on. But wider issues are involved, which, so far as I know, have never been explained.

The general information given in virtually all shade-cards is that "dyeing is done at quoted temperatures for 30-60 or more minutes" and so forth. And yet all chain-warps, and pieces continuously dyed, are in contact with the dye liquor only for a matter of seconds.

I well remember colleagues of mine in the former I.G. in Germany interesting themselves in the chain-warp-dyeing methods used in Great Britain and doubting the fastness to be obtained by short immersions. As a result of discussions it was decided to obtain cuttings from dyed warps (chain-dyed in bulk) in pale, medium, and dark shades. These dyeings were then matched, with dyes identical with those used on the warp, on 10-g. hanks, using the same cotton but dyeing by the normal 60-min. or longer process. Subsequently a full range of fastness tests was made. The fastnesses of the bulk-chain-dyed warp were identical with those of the laboratory-processed yarn.

I am not aware that so very interesting a fact has ever been explained. The only comment I heard or made was that the liquor ratio in the dyeing of chain-warps was excellent. Such comment I regard as too generalised and incomplete. Perhaps some of our members may be able to explain or decide to research on the problem. And is there not a moral somewhere which we all seem to have missed all this time? By the way, when processing union (wool-cotton) cloth in medium depths the immersion in the dye liquor never exceeded 20-25 sec., but the process I used has never been published.

HERBERT A BRASSARD

136 GROVE HALL COURT
HALL ROAD
LONDON N.W.8

19th November 1956

Notes

Meetings of Council and Committees December

Council—5th
Finance and General Purposes—5th
Diplomas—5th
Terms and Definitions—7th
Publications—18th
Textbooks and Monographs Subcommittee—18th

Election of Fellow

At the meeting of Council held on 5th December 1956 the following was elected a Fellow of the Society—

Thomas Flanagan
(Liverpool; Dyehouse Manager, British Enka Ltd.)

Miss Esmée Smith

Honorary Secretary of the West Riding Section

Miss Esmée Smith has been elected Honorary Secretary of the West Riding Section in succession to Mr. G. E. Styan, who has been warmly thanked for his services by the Chairman of the Section, Mr. C. W. Green. Miss Smith is a daughter of Mr. Fred Smith, Immediate Past President of the Society. Her address is Cliffe House, Bruntcliffe, near Leeds.

The Journal in Forty-five Countries

The Society's *Journal* has appeared every month for seventy-two years, giving not only an account of the activities of its Sections and Branches, but

providing a medium for the publication of scientific and technical communications, together with abstracts from British and overseas publications covering most of the matters which are interesting to those engaged in the science and technology of dyes and dyeing in all branches of industry.

Some notion of the modern growth of the Society, with its widely used services, can be drawn from the international nature of its membership, which is spread over thirty-eight countries. The *Journal* circulation covers forty-five countries, which demonstrates the growing significance of the important part played by the *Journal* all over the world.

Besides the British Isles, the countries included are—

Africa	Formosa	Norway
Argentina	*France	Peru
*Australia	*Germany	Philippines
Austria	Greece	Poland
*Belgium	Haiti	Roumania
Brazil	*Holland	Siam
Bulgaria	Hungary	South Africa
British West	Israel	*South America
Indies	*India	*Spain
*Canada	Indonesia	Sweden
*China	*Italy	*Switzerland
Czechoslovakia	*Japan	Turkey
Denmark	Lebanon	*U.S.A.
*Egypt	Malaya	U.S.S.R.
*Finland	New Zealand	*Yugoslavia

Among these, the principal users are those countries which are marked with an asterisk.

Prices of Abstracts and Reprints

In conformity with the increased price of the *Journal*, the charge for abstracts printed on only one side of the page is now £2 0s. 0d. per annum. The new prices for reprints are 3s. 4d. per single copy, 16s. 0d. per dozen copies of papers occupying up to 8 pages, and 22s. 6d. per dozen copies of papers occupying more than 8 pages.

Library—Catalogue of Books and Periodicals

A list of books and periodicals held by the Society has been prepared, and mimeographed copies are available to members on application to the Editor. Members may borrow most of these and other books reviewed in the *Journal*, and also the periodicals abstracted.

The attention of members is again drawn to the privilege enjoyed by them of borrowing books from the extensive collection in the Library of the Chemical Society (see p. 2).

Association of British Chemical Manufacturers Appointment of New Director

The Council of the Association has appointed Mr. George Brearley, B.Sc., F.R.I.C., M.I.Chem.E., as Director of the Association in succession to Mr.

J. Davidson Pratt, who is retiring in June 1957. Mr. Brearley has, since April 1953, been a partner of Cremer and Brearley, Consulting Chemical Engineers. From 1926 to 1953 he was employed by Brotherton & Co. Ltd., and was a director of that company from 1938 and Managing Director from 1946 until his resignation in 1953. Mr. Brearley's A.B.C.M. activities have included membership of Group D and participation in Chemical Works Safety Conferences. In order to allow for a period of overlap, Mr. Brearley will take up his appointment with the Association on 1st April 1957.

Interdependence of Technical Societies and Industry

In an interesting lecture to the Eastern Townships Textile Association in Quebec (*Canadian Textile J.*, 73, 28 (19th October 1956)) Dr. L. J. Sheps states that the membership of a technical group must represent breadth of experience and yet sufficient closeness of interest to ensure a satisfactory programme of activities for the majority of members. While the formal objects may be very wide, so that the group is permitted to do whatever it may desire, its actual activities are best kept within fairly close limits, so that other groups may be formed in related fields of interest: different societies should be complementary rather than competitive. Advancement of knowledge improves industry, directly or indirectly, but in return industry contributes the time of honorary officers and members of committees, and supports them with office services and even travelling expenses. Officers should try to avoid keeping within cliques, which, however, may have some practical value in canalising thinking. Committees should be small, but work should be spread among as many members as possible. Work in a technical society may be a valuable training for increased responsibility in industry.

Hydrosulphite Fire Danger

It appears that sodium hydrosulphite is being sent through the post. The Post Office authorities regard it as a dangerous substance, and any person who sends, attempts to send, or procures to be sent by post a packet containing a dangerous substance is liable to prosecution.

Hydroxymethyldimethylhydantoin

This compound, abbreviated to "MDMH", is now being produced commercially in the U.S.A. by the Glyco Products Co. Inc., of Williamsport, Pa. It is recommended for use as an almost odourless formaldehyde donor, and is said to contain 19% combined formaldehyde which is capable of liberation. It insolubilises proteins such as gelatin and casein, and can be employed for modifying starch, textiles, fur, leather, paper, and wood.

Dynel Filtration Felts

A new type of non-woven felt composed of dynel acrylic fibre with a proportion of thermoplastic binder fibre has been designed by the American Felt Co. specifically for the filtration of dyes and pigments. It is known as Dynel Windsor Felt, and is claimed to retain very fine particles without clogging.

The Telomerisation* Reaction and Nylon Manufacture in the U.S.S.R.

In 1948 Joyce, Hanford, and Harmon (*J. Amer. Chem. Soc.*, **70**, 2529 (1948)) discovered the radical-initiated telomerisation* of ethylene with chlorohydrocarbons. This reaction has since received much study, and in the Soviet Union, where great possibilities for the industrial exploitation of this reaction are envisaged, a school of chemists led by A. N. Nesmeyanov is carrying out an extensive programme on its use for the synthesis of a great variety of substances, many of which are listed in a review of this work by Nesmeyanov in *Quarterly Reviews Chem. Soc.*, **10**, 330-370 (1956).

Telomerisation of ethylene with carbon tetrachloride gives $\alpha\alpha\omega\omega$ -tetrachloroalkanes $\text{Cl}[\text{CH}_2]_{2n}\text{CCl}_2$ containing an odd number of carbon atoms, the predominant value of n being determined largely by the relative amounts of the reactants; members of this series containing up to fifty-one carbon atoms have been isolated and characterised. These compounds are readily converted into "odd" ω -amino carboxylic acids $\text{H}_2\text{N}[\text{CH}_2]_{2n}\text{COOH}$, which can be converted into nylons. Enant (7-nylon) fibre, which is now being developed in the Soviet Union, is manufactured in this way (cf. *Faserforsch. und Textiltech.*, **7**, 422-424 (Sept. 1956)); though in many ways similar to 6-nylon fibre, Enant has a higher melting point and is superior in other respects (see *J.S.D.C.*, **72**, 304 (June 1956)). The same telomerisation products can be converted also into ω -cyano acids, which can be reduced to "even" ω -amino acids and can be hydrolysed to "even" alkanedioic acids (e.g. adipic acid); "odd" alkanedioic acids can be obtained by oxidation of the ω -hydroxy acids obtained by the complete hydrolysis of the telomerisation products. All these amino acids and dicarboxylic acids can be used in the preparation of nylons.

A.E.S.

*Telomerisation is a word coined by American workers for the process by which polymers of low molecular weight (low D.P.) are formed in a polymerising system by chain transfer from the solvent or added retarding agent. Thus in carbon tetrachloride chain growth in the free-radical polymerisation of vinyl monomers is stopped by addition of Cl and CCl_2 groups to the ends of the chains.

New Dyemaking Plant in India

At Kalyan near Bombay a new plant for the manufacture of dyes, fluorescent brightening agents, and pigments has been opened by Amar

Dye-Chem Ltd. of Bombay and Sterling Drug Inc. of New York. It is reported to be the first plant in India for the manufacture of azoic components.

Research on Food Dyes

According to *Food and Drug Research* (3, 1 (July 1956)) a committee appointed by the National Academy of Sciences in the U.S.A. has recently reviewed the research programme of the Food and Drug Administration. It recommends continuation and extension of present fundamental investigations on such problems as improved analytical procedures, the metabolic fate of certifiable dyes, and their toxicity and carcinogenicity.

The report of the committee reveals that fifteen of the nineteen food dyes certifiable until recently have been studied since 1945; of these several are "distinctly toxic when fed to rats at levels as low as 0.25% of an otherwise standard diet"; FD&C Orange No. 1, FD&C Orange No. 2, and FD&C Red No. 32 have been removed from the list of certifiable food dyes (except that the latter has been continued by special act of Congress for use on oranges); FD&C Reds No. 2, 3, and 4 and Yellows No. 1, 3, 4, and 6 are being intensively examined, Yellow No. 6 and Red No. 4 for carcinogenic potentiality. The decision of the court on the appeal against the de-listing of certain FD&C colours has not yet been reported.

In the light of the official position in the U.S.A. that the law does not grant authority to establish tolerance levels for "harmless and suitable" coal-tar dyes, special interest attaches to the statement of the *ad hoc* committee that it "feels compelled to indicate that certification of a compound as 'harmless and suitable for use' in foods, drugs, and cosmetics as required under the present law is unrealistic unless the level of use is specified." It has been the contention of the American food dye industry that the establishment of appropriate tolerance limits regulating the normal use of food dyes might remove the need to decertify them.

In view of the furore over the re-evaluation of what have been legally established as "harmless" food dyes in the U.S.A. the status of these dyes in other countries is of particular interest. An extensive tabulation of some 102 dyes, one or more of which have been permitted or proposed in 33 different countries (Sadini, V., *Rivista italiana d'Igiene*, **15**, 399 (1955)) shows the wide disparity existing in international practice: for example, more than 25 countries permit the use of FD&C Blue No. 2, Red No. 2, and Yellow No. 5; but less than 10 permit the use of Red No. 32, Blue No. 1, Green No. 3, Orange No. 2, Red No. 32 and Yellows No. 3 and 4.

New Books and Publications

The Selected Papers of Dr. Toshio Maki on Vat Dyes

Compiled and edited by Y. Nagai and S. Kato. Pp. viii + 248. University of Tokyo. 1955.

Professor Dr. Toshio Maki, Dr. Eng., whose death was announced recently, was one of the most eminent of researchers in the field of dye chemistry. He was born in 1895, and died in October 1954, after a notable career in which he made many important contributions to the chemistry and technology of dyes. The present volume lists 98 papers that carried Maki's name and reprints a selection of 70 of these in full. The majority of the papers are in Japanese, but 5 are in English and 5 in German, and many have been mentioned in the abstracts of the Society's *Journal*. The scope of the present volume is shown by the number and variety of papers which have been brought together. These include publications on the indanthrone series (18); benzanthrone (33); pyrazoloanthrone (5); acenaphthene (6); amide, imide, carbazole, and acridone series (5). As the title indicates, all the papers are concerned with vat dyes, and all of these are of the anthraquinone type. W. BRADLEY

Anorganische Textilfaserstoffe Asbest, Glas, Schlacke, Gestein, Metall (Gewinnung, Eigenschaften, Verarbeitung, Veredlung, Verwendung)

Edited by W. Bobeth, W. Böhme, and J. Techel. Pp. 570. Berlin: VEB Verlag Technik. 1955. Price, DM 35.80.

As stated in its subtitle, this work sets out to cover the origin, properties, manufacture, finishing, and uses of fibres from asbestos, glass, slag, minerals, and metals. Such an aim could be carried out only by a team, and it is not surprising, therefore, to find that the editors have had the assistance of a further seven collaborators.

After a brief introduction to fibres in general, the economics and the manufacture of inorganic fibrous materials are discussed at some considerable length in the second chapter. This is followed by another long and detailed chapter on the properties of the fibres—chemical structure, physical and chemical properties, and the analysis of fibre mixtures. Descriptions of the spinning, weaving, dyeing, and finishing processes for the different fibrous materials fill the next three chapters.

In the short review of the potential uses which follows, some idea can be gained of the value of inorganic fibres. No doubt the importance of the subject to these trades has prompted the authors to include a chapter (which is very heavy going for the layman) on the health hazards involved. A final chapter deals with dust and its elimination, which is concise and of general interest.

To have collected all this information in one volume would have been a signal service in itself, but the volume is doubly valuable in that it is the first comprehensive work in a field in which the documentation is very sparse. For these reasons there appears to be little doubt that the book will be welcomed by technologists handling these

materials. Its potential interest is wider than this, however. To those interested in the fibres which must be termed, in this context, organic, it will evoke no little surprise to find so much activity in industries which are closely related, but are almost entirely ignored by them. Readers of the *Journal* will find not only much that is familiar in the section on dyeing, printing, and finishing, but also techniques which are completely different, and specific to the fibre concerned. This raises the question of the value of the book to teachers. There would appear to be useful material to be gleaned from the similarity and the dissimilarity between inorganic and organic fibre technology, in the same way that useful analogies can be drawn between the natural and the man-made organic fibres.

This comparison should be facilitated by the fact that the authors themselves have included organic fibres for comparison at various points in the text, particularly in Chapter 3, dealing with fibre properties. In Chapter 6, Section 1, the improvement of fibre properties again furnishes possibilities of this kind. The much wider use of glass fibre, the appearance of new metallic effect threads, and the increasing emphasis on fireproof textiles, to quote only a few examples, may very well require the complete textile technologist to have more than a passing acquaintance with inorganic fibres.

The diagrams in the book are useful, and the photographs that are used have a surprising clarity. A list of 434 references is given.

S. R. COCKETT

Wool Wax Chemistry and Technology

By E. V. Truter. Pp. xi + 368. London: Cleaver-Hume Press Ltd. 1956. Price, 60s. 0d.

The research work carried out by Dr. Truter at Leeds University during several years has been the subject of numerous papers which have appeared in research journals, and the publication of this book on wool wax further demonstrates the author's wide knowledge of many aspects of this specialised branch of chemical technology.

In the preface, Dr. Truter presents his book as a first critical account of the chemistry and technology of wool wax, a claim which can be fully justified by the absence of other textbooks dealing specifically with wool wax and related products.

The book does not claim to provide an exhaustive bibliography to the patent and technical literature, but references are very extensive, and add appreciably to the review published by Gillespie in 1948 (*J. Textile Inst.*, 39, p45). Adequate author and subject indexes are provided.

The scope of the book covers the subject to the extremes, beginning with the study of the wax on the fleece, where it is inseparably bound up with a study of wool and suint, through recovery, purification, processing into derivatives, chemistry, and ultimate uses, including a well presented account of the recovery of wool grease products at Bradford Corporation Sewage Works. It is

interesting to note in this chapter that Dr. Truter is under no impression that grease recovered from sewage effluent is used in cosmetics, as is sometimes believed.

Each aspect is treated very thoroughly, often including pages on relevant theory, such as that relating to emulsions, foams, detergency, saponification kinetics, etc. Perhaps the book is rather unbalanced in this respect, as no less than 40 pages (almost one-ninth of the book) are devoted to the basic theory of clathrate compounds. Moreover, 29 pages are given to the derivation of the chemical structure of lanosterol and related compounds, whilst the equally or more important corresponding history of the structure of cholesterol is not dealt with anywhere near so fully. However, the chapter on the isolation and determination of cholesterol is characteristically thorough, and on the whole the omissions in the book are not vital. On the other hand, the processing of wool wax into pharmaceutical lanolin is perhaps sketchily described, and whilst the description of the production of wool-wax alcohols is detailed enough, the processing into B.P. quality is not mentioned. This is rather regrettable, as pharmaceutical lanolin and wool alcohols are probably the most important products of wool wax. No doubt, however, such information is understandably difficult to obtain from the few manufacturers in the field.

The book can be described as very well presented in a good type of print with bold section headings in each chapter, facilitating both indexing and reading. Typographical errors are few, although minor mistakes are apparent; for instance, a reader will have doubts about the description of raw-wool scouring in which the wool is said to remain 10-15 min. in each bowl of a scouring set. Also in Table V.8 on p. 135 the ash content of a good-quality acid-cracked wool grease is given as 1%—probably an error for 0.1%, as such a figure would be nearer the mark.

To summarise, Dr. Truter's book, although rather expensive, will doubtless be a valuable and often referred-to possession of many owners, in industries varying from wool-combing to hormone production.

E. W. CLARK
R. HULLAH

Paint and Varnish Manufacture

Edited by H. W. Chatfield. Pp. 440. London: George Newnes Ltd. 1955. Price, 35s. 6d.

This is a compilation of essays by as many as thirty-three contributors. It is therefore reasonable to expect some unevenness of treatment, but the effectiveness of the work as a whole would be enhanced if the treatment were a little less uneven than in fact it is. The claim that this book is "a complete survey" of the paint industry is warranted only insofar as the main aspects of the technology are included, but it cannot be claimed that they are all fully covered, for there are important technical features which are touched upon but lightly. Part 1 covers raw materials; Part 2 (about one-third of the text) deals with manufacture, which the title of the book suggests as the main subject: indeed,

only one section in Part 2 (about sixty pages) deals with paint and varnish manufacture as such.

There is much useful information, many of the contributors being well known specialists in their several fields. For the most part, academic language is happily avoided, as befits a book intended mainly for "the student, the uninitiated, and technologists with varying amounts of experience". It may be properly regarded as a handbook for students and as such a useful addition to technical literature in that class. Indeed, the student's handbook idea was manifestly in the minds of many of the authors, who, with limited space, have treated their subjects briefly, given typical compositions with brief descriptions of properties and applications, and wisely referred the reader to more detailed works for more comprehensive study. Unfortunately, the references are not uniform: quite often the title of a particular journal is abbreviated in a number of forms; this might be remedied in future editions. There is a great deal of useful data in tables and an adequate index.

S. H. BELL

Handbuch der chemischen Untersuchung der Textilfaserstoffe

Zweiter Band

Chemismus, Eigenschaften und Einsatz der textilen (nicht veränderten) Faserstoffe und ihre Prüfung

By H. M. Ulrich. Pp. xxvii + 744 + 113 Fig. Vienna: Springer-Verlag. 1956. Price, DM 110.00.

Volume II of this German "Handbuch" follows the pattern set by Volume I (cf. J.S.D.C., 70, 359 (1954)), but with more than twice the number of pages.

To translate the title of the whole series by "handbook", when this volume alone weighs nearly 4 lb., would be a false use of a word which the *Oxford English Dictionary* defines as "a small book or treatise such as may be held in the hand."

As with the first volume, Dr. Ulrich's second one on the chemical investigation of textile fibres provides a great mass of detailed information. That it appears only two years after the first volume is a testimonial to the author's industry and knowledge. The present volume covers the chemistry, properties, and textile uses of fibres and their testing. In addition to the general text, there are useful bibliographies in the different sections scattered through the book. As with the first volume, this one has no index, and so one must browse through the book to find whether or not it contains any particular item. This makes the book difficult to consult, which is a grave defect in a work of reference.

Examination of a few sections taken at random shows that the treatment, though extensive, is not exhaustive, and a reader would lack much relevant information if he depended upon it alone. Furthermore, as with so many compendious works, the treatment of the data given is not critical and must be used with caution. A present defect is the use of cross-references to the later volumes, which may not be available for some years. A critical reader must doubt the wisdom of these principles upon which the work is constructed.

One presumes that an index will be provided with the final volume when it is published, and it will not be till then, when the work is complete, that it will be possible to consult it readily and to assess its value properly. By that time, much of the topical matter in the earlier volumes will be out-dated. When one has admitted the limitations mentioned above, still the book has merit and will prove helpful to workers in the field, since, when they refer to it, it will give them a useful lead occasionally.

J. M. PRESTON

Wool Research

Volume 2

Physical Properties of Wool Fibres and Fabrics

Pp. vi + 234. Leeds: Wool Industries Research Association. 1955. Price, 30s. 0d.

Volume II in the W.I.R.A. series *Wool Research* has made a welcome appearance. Like its companions, it does not claim to be a complete textbook on its subject, but rather an extended report on the research done by the Association on the physics of wool fibres and fabrics. The extent of this limitation of its scope and value varies somewhat throughout the book, since the Association's contribution to some topics is very comprehensive in itself, while mention of other parallel work is usually made.

The work discussed comprises the physics of wool-moisture systems, the warmth of clothing, the water-repellency of fabrics, the electrical properties, the elastic and plastic properties, and the friction of wool. The book ends with a chapter on colour. In each chapter the technological applications of the research are pointed out, so that the reader in industry may be encouraged to struggle with the scientific ideas and argument.

The chapters make varying demands upon previous training in physics, but the book is not difficult to a reader who is prepared to face conclusions expressed in simple graphs and equations and then amplified in words. Mathematical developments of theory are generally avoided. The graphical illustrations are excellent and numerous, and the general standard of presentation is very good.

The chapter on friction is particularly complete, and those on air-flow and the warmth of fabrics very thorough. Unfortunately for readers of the *Journal*, the chapter on colour is not the best in the book, being too closely condensed to be easily followed by a novice, who might, incidentally, be further confused by the use of three different units of wavelength in the four pages 188-191. Work on fastness testing and on the theory of dyeing has been omitted from this volume.

The book is valuable in collating much scattered material and presenting it in assimilable form. It may serve as a source book for future textile engineers and physicists, and its appearance will strengthen courses in textile science.

W. J. ONIONS

Studien an einem neuzeitlichen Kreuzpultrockner für Bastfasergarne mit Wiederbefeuchtungszone

Forschungsberichte des Wirtschafts- und
Verkehrsministeriums Nordrhein-Westfalen No. 185

By W. Rohs and G. Heller. Pp. 39. Cologne:
Westdeutscher Verlag. 1955. Price, DM. 10.70.

This report deals with the drying of flax yarns on cheese after a wet-spinning process. Experiments were conducted with a three-zone Jaeggli drier in which hot air is blown through cheeses on perforated tubes. The effects of changes in temperature and humidity of the drying air were studied. It was found that, contrary to general belief, yarn quality did not suffer when air of low humidity was used to speed drying; quality was, in fact, improved. The rate of drying was found to be appreciably increased by increase in the extent of tube perforation and decrease in the ratio of external to internal diameter of the cheese; in addition, it was found that the design of the individual supports for the cheeses could affect rate of drying.

The possibility of reconditioning the cheeses with moist air in the last zone was also studied. It was found that, without appreciably increasing the time the cheeses spent in the drier, it was possible to bring them to a uniform moisture content of 7% and thus avoid the very slow reconditioning at room temperature necessary after the usual drying process.

The authors note that drying in a multiple-zone apparatus is more complicated and requires better control of temperature and humidity than an ordinary drying process; they suggest that some difficulties would be removed if drying and reconditioning zones were made completely separate installations.

R. F. ELLIS

Die Messung von Gewebetemperaturen mittels Temperaturstrahlung

Forschungsberichte des Wirtschafts- und
Verkehrsministeriums Nordrhein-Westfalen No. 199

By the Textilforschungsanstalt Krefeld. Pp. 36.
Cologne: Westdeutscher Verlag. 1955. Price,
DM. 10.90.

After a general discussion of conventional contact thermometers and the reasons for their unsuitability for measurements of fabric temperature, the development is described in some detail of a radiometer (utilising copper-Constantan thermocouples) suitable for the measurement of fabric temperatures in the range 45-250°C. and having an accuracy of $\pm 1.5^\circ\text{C}$. and a time-constant of response of about 1 sec. The instrument can be made portable or can be installed in a heating or drying zone. In the latter case the difficulty of maintaining the reference thermocouple-junctions at a fixed temperature is overcome by surrounding the measuring elements with a jacket containing a liquid which is kept at boiling point. The vapour is condensed in a second chamber, and the condensate returned to the jacket. This principle can be used to produce a compact and self-contained measuring head so long as a liquid can be found

which has a boiling point above the temperature of the heated zone, and which is suitable in other respects. If this condition cannot be satisfied, the measuring head must be made less compact and less mobile by separating the thermostating jacket and the condensing chamber and placing the latter outside the heated zone; alternatively,

"windows" into the heated zone would have to be available.

It is found that, with the exception of metallised goods, fabrics of cotton, rayon, or nylon behave as black-body radiators irrespective of their finish, so that one calibration serves for all fabrics.

R. F. ELLIS

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Extracting Liquid from Wet Material

Bowaters Development & Research

BP 758,732

A hollow rotatable drum is divided into sections communicating with the bore of the drum and has a suction box mounted internally, the walls of which bound a pressure-equalising passage. This rotary drum reduces the work required to maintain the suction zone at a reduced pressure and can be used to filter slurries or to extract water from wet felts.

K.W.

Winding Mandrels having Suction Means

Bowaters Development & Research

BP 756,355

A suction box is fitted radially to an apertured pad placed in a slot in the mandrel surface, where the radius of curvature of the pad is less than that of the mandrel surface, and the underside of the pad is longitudinally recessed to provide a suction passage, so that thick and heavy webs are anchored even when wound under considerable tension.

K.W.

Coating of Webs in Web-feeding Machines

Bowaters Development & Research

BP 756,356

A shielding device, consisting of two guide plates, is combined with a coating roll so as to interrupt the coating process and to support the web clear of the roll and shield it from the application of coating substance whilst the web continues to travel.

K.W.

Raising Machine giving Good Orientation of the Nap

Aa Be Wollenstoff- en Wollendekenfabrieken

BP 760,894

Preboarding Machine

J. W. Lightburn & Son

BP 759,525

A machine of the horizontal-chamber type in which the form carriage, after emerging from the chamber along a track, is pivoted to move transversely along a single rail, thus enabling another carriage to be introduced. The provision of a second carriage for the transverse movement, as in earlier machines of this type, is thereby eliminated. A vertically slidable chamber door allows the volume of the chamber to be reduced.

G.E.K.

Sock-drying Form

Crayonne

BP 758,258

A plastic framework form in which the leg and heel portions are connected by pivot to one arm of a U-shaped resilient foot member. During mounting of the sock the free, spring-like arm of the foot member is constrained, but the recovery action is utilised in adapting the framework to the foot-size of the garment.

G.E.K.

Garment Press

Bishop David Freeman Co. and Davis Press Pad Co.

BP 759,865

A device for fixing to the head of the press to ensure uniform distribution of the steam over the garment.

C.O.C.

Printing or Coating Metal Sheets

Metal Box Co.

BP 758,719

II—WATER AND EFFLUENTS

Transformations of Carbon Compounds by Micro-organisms

W. J. Nickerson

Ind. Eng. Chem., 48, 1411-1420 (Sept. 1956)

Some typical reactions which take place in microbiological decomposition processes are described. Exo-

cellular transformations discussed include the decomposition of cellulose, chitin, and keratin, and the oxidative degradation of aromatic substances such as lignin. Dissimilative reactions involved in fermentation and oxidative metabolism are discussed as examples of intracellular transformations.

W.K.R.

Biological Transformations of Nitrogen Compounds

C. C. Delwiche

Ind. Eng. Chem., 48, 1421-1428 (Sept. 1956)

The mechanisms by which nitrogenous compounds are decomposed by micro-organisms under aerobic and anaerobic conditions are reviewed and discussed with particular reference to sewage disposal.

W.K.R.

Transformations of Sulphur by Micro-organisms

R. L. Starkey

Ind. Eng. Chem., 48, 1429-1437 (Sept. 1956)

The various reactions by which sulphur compounds are transformed by micro-organisms are reviewed. Two principal products are formed—sulphide under anaerobic conditions and sulphate under aerobic conditions.

W.K.R.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Fatty-acid Esters of Sucrose—Surface Activity of Monoesters

L. Osipow, F. D. Snell, D. Marra, and W. C. York

Ind. Eng. Chem., 48, 1462-1464 (Sept. 1956)

Laboratory evaluation of the surface-active properties of sucrose monoesters (laurate, myristate, palmitate, oleate, and stearate) indicates that they are good detergents, and when used in conjunction with a more lyophilic emulsifier, e.g. glycerol monostearate, they are good emulsifiers. They have low toxicity. Data on physical properties are recorded.

W.K.R.

Fatty-acid Esters of Sucrose—Methods of Preparation

L. Osipow, F. D. Snell, W. C. York, and A. Finchler

Ind. Eng. Chem., 48, 1459-1462 (Sept. 1956)

Sucrose (3 moles) reacts with fatty-acid methyl ester (1 mole) and potassium carbonate (0.1 mole catalyst), in sufficient dimethylformamide or dimethyl sulphoxide to dissolve the reactants, on heating at 90-95°C. and 80-100 mm. pressure for 9-12 hr. Methanol is stripped off, much of the solvent is removed by distillation, and the residue is dried under vacuum, providing the sucrose monoester in 90% yield. The diester is prepared in 90% yield by reaction of 2 moles of methylester and 1 mole of sucrose under similar conditions. The products may be purified by partitioning the crude residue between aqueous salt solution and n-butanol. Analytical procedures for sucrose esters are described in detail.

W.K.R.

Micelle Formation in Solutions of Isomeric Detergents

D. B. Ludlum

J. Phys. Chem., 60, 1240-1244 (Sept. 1956)

Measurements of electrical conductivity and optical turbidity are reported for three isomeric dodecylbenzene-sulphonates in water. Critical micelle concentrations and micelle sizes are obtained. It is concluded that an increase in hydrophobic nature lowers the critical micelle concentration and increases the micelle size, in agreement with recent theories of micelle formation.

W.R.M.

Equilibrium Dialysis of Soap and Detergent Solutions

H. B. Kleven and C. W. Carr

J. Phys. Chem., **60**, 1245-1249 (Sept. 1956)

Dialysis measurements have been made on a series of anionic and cationic soaps and detergents. Equilibration across Cellophane membranes was observed in all cases of salt-free and polar-hydrocarbon-free detergent solutions. Long-chain alcohol and amine additives and changes in ionic strength markedly increased the time for equilibrium to be reached. The results suggest that earlier measurements and interpretations as to critical micelle concentrations and micelle formation based on them require reconsideration.

W.R.M.

PATENTS

Detergents of Polyoxyethylene-ether Type

Atlas Powder Co.

BP 759,854

The product obtained by treating 10-20 mol. of ethylene oxide with 1 mol. of a saturated branched-chain alcohol of 11-15 C of the type produced by the OXO synthesis from $C_{10}-C_{14}$ olefinic polymers of propylene or isobutylene have valuable surface-active properties and are especially useful as detergents.

C.O.C.

Detergent Compositions

L. McDonald

BP 759,136

The properties of synthetic detergents and of their mixtures with soap are considerably improved by addition of a little of a water-insoluble cellulose ether.

C.O.C.

Detergent Compositions

Monsanto

BP 759,143

Ionic detergents form complexes with alkylmonocamines (Alk of > 6 C) and such mixtures have in many cases much greater detergent power than the ionic detergent.

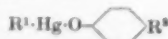
C.O.C.

Bactericidal and Fungicidal Arylmercury Compounds

Ward Blenkinsop & Co.

BP 758,730

Compounds of formula—



(R^1 = monocyclic arylhydrocarbon group; R^2 = Alk of 4-12 C), e.g. phenylmercuric *p*-(1:1:3:3-tetramethylbutyl)-phenoxide, are readily soluble in hydrocarbon solvents and are used for the rotproofing of paints, ropes, etc.

C.O.C.

Dyeing Assistants and Stripping Agents

Gy

BP 759,331

The condensates of mineral-acid esters of saturated fatty alcohols of 16-18 C with excess of ethylenepolyamines when treated with 15-20 mol. of ethylene oxide yield water-soluble waxy products useful as levelling agents, particularly for milling and metal-complex acid dyes containing no sulfo groups. They can also be used as stripping agents for wool dyed with acid dyes.

C.O.C.

Levelling and Stripping Agents for Metallised Dyes

American Cyanamid Co.

USP 2,723,178

Compounds of formula $R^1\text{-CO-NH-CH}_2\text{CH}_2\text{-NR}^2\text{R}^3\text{R}^4\text{Y}$ (R^1 = aliphatic radical of > 6 C; R^2 and R^3 = Alk; R^4 = Alk, hydroxyalkyl, aralkyl, or unsaturated aliphatic hydrocarbon radical; Y = anion), e.g. γ -stearamidopropyl-hydroxyethyl-dimethylammonium chloride, are excellent levelling agents for use with metallised dyes. They form no objectionable scum in the dyebath. Their use enables organic acids to be used instead of sulphuric acid. They can also be used as stripping agents for metal-complex dyes.

C.O.C.

Paper-sizing Materials

Monsanto

BP 755,149

The size produced by reaction of rosin with an aliphatic olefinic dicarboxylic acid or its anhydride, e.g. maleic anhydride, at 200-260°C. and an absolute pressure of 15-200 mm. Hg is substantially free from rosin oils, does not cause appreciable foam formation, and reduces the tendency of the pulp solids to float on the surface of the suspension.

K.W.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS**A Historical Outline of the French Dye Industry**

A. Fresco

Rev. Textile, **55**, 336-339 (1956)**The Disperse Dyes—Their Development and Application**

R. K. Fourness

J.S.D.C., **72**, 513-527 (Nov. 1956)

A review with 64 references.

Colour Theory of Organic Compounds—I and II

B. I. Stepanov

Zhur. fiz. khim., **29**, 2173-2184 (1955); **30**, 34-49 (1956);*Chem. Abs.*, **50**, 13592 (10 Oct. 1956)

I—Light absorption in the visible spectrum by organic compounds is connected with the presence of open or closed systems of conjugated double bonds in the molecules. Addition of polar groups to the conjugated systems displaces the absorption spectrum range towards the longer wavelengths and increases the absorption intensity. An ionisation of the molecules which increases the electron-donor or electron-acceptor properties of the group added is accompanied by shifting of the absorption spectrum toward the longer-wavelength region and increases the absorption intensity; destruction of the electron-donor properties of the electron-donor replacements has a reverse effect.

II—Reduction of the constant electron displacement (i.e. not depending on light effects) in a conjugated-bond system between polar substituents or in some parts of the molecule and caused by introduction of new substituents, closing of rings, and similar changes in the molecule of an organic compound displaces the light absorption into the shorter-wavelength range. Changes in the molecular planarity, resulting in partial or complete separation of a conjugated system, is accompanied by displacement of absorption toward a shorter wavelength and lowering of the absorption intensity. Complex formation with metals results in a deeper colour when the co-ordination bond with the metal is formed with an undivided atomic electron pair which is part of the system causing the coloration.

C.O.C.

Steric Factors in the Hydrolysis of Potassium Chlorodinitrobenzenesulphonates

J. Radell, L. Spialter, and R. C. Jamagin

J. Org. Chem., **21**, 1032-1033 (Sept. 1956)

Sulphonation of chlorobenzene followed by nitration gives a product containing mainly 4-chloro-3,5-dinitrobenzenesulphonic acid (I), but also some of the 2-chloro isomer. This remains in the mother liquor on recrystallising the K salt or can be removed as 2,4-dinitrochlorobenzene on steam-distillation of an aqueous sulphuric-acid soln., I remaining unchanged.

H.E.N.

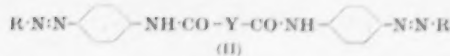
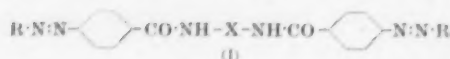
Relationship between Structure and various Properties of Azo Dyes containing Amide Groupings

B. M. Krasnovitskii, R. M. Matskevich, and

T. M. Nikishova

Doklady Akad. Nauk S.S.S.R., **108**, 91-94 (1 May 1956)

Previous work on azo dyes derived from benzamide and containing one amide (CO-NH) grouping (see *J.S.D.C.*, **72**, 386 (Aug. 1956)) is now extended to analogous dyes containing two amide groupings; the results on substantivity for cellulose and colour are interpreted along the same lines as before. The dyes studied are of types I and II, in which R is an H-acid residue (alkaline coupling). In one pair of isomers, I_0 and II_0 , X and Y are absent, the amide groupings being directly linked together. In other dyes $X = Y = III$, $X = Y = IV$, $X = CO$, and $Y = V$. II_0 is much more substantive than I_0 , but for the other pairs of isomers the difference in substantivity is only slight. When $X = Y = III$, II is more substantive than II_0 , and I is much more substantive than I_0 ; but the introduction of a second benzene ring ($X = Y = IV$) produces no further effect. Introduction of $X = CO$ into I_0 results in almost complete loss of substantivity (an unbroken conjugated chain can no longer be formed), and the introduction of $Y = V$ into II_0 also results in a great reduction in substantivity (contrast $Y = IV$), which is attributed to steric disturbance of nuclear coplanarity in the diphenyl residue (this results also in a considerable lightening of the colour). Also, the acylaminoanthraquinone vat dyes derived from 1-aminoanthraquinone (2 mol.) and the dicarboxylic acids (1 mol.) based on the radicals IV and V are compared, and the former is found to have a much higher substantivity than the latter.



Investigations on Colour Development. XV—Semiquinones as Colour Developers

W. Brune and U. Kronacher

Z. wiss. Phot., **50**, 78-95 (1955);
Science et Ind. phot., **27**, 280-281 (1956);
Chem. Abs., **50**, 12708 (25 Sept. 1956)

The semiquinone of *p*-aminodiethylaniline produces more dye for a given amount of Ag development than does the parent compound.

C.O.C.

Azo Dyes. VII—Azo Dyes containing the Thiolglycolic Acid Group

K. Murata and K. Harada

Bull. Fac. Engr. Hiroshima Univ., **5**, 13-18 (1956);
Chem. Abs., **50**, 13445 (25 Sept. 1956)

5-Chloro-2-aminophenylthioglycolic acid has been coupled with various azo components, and the resulting dyes have been examined for dyeing properties on animal fibres.

C.O.C.

Formazan Dyes

H. Ziegler

Compt. rend 27^e Congr. intern. Chim. ind. (Brussels), **3** (1954);
Industrie chim. belge, **20**, Spec. No. 673-678 (1955);
Chem. Abs., **50**, 13782 (10 Oct. 1956)

A new method for obtaining excellent yields of *meso*-aryloformazans, 2:5-X(CH₂SO₂)C₆H₃NH:N:CAr:N:N-C₆H₄-Y-2 (I) directly couples Ar-CHXY (X and Y = electron-attracting substituents, e.g. COOH, COOR, CO-NH₂, CN, CHO, CO-R) in succession with two diazotised amines or aminophenols. In the classical synthesis of I, the less readily accessible arylhydrazines are required. Hitherto unobtainable Cu and Ni complexes of *oo'*-dihydroxy-*meso*-aryloformazans can be prepared by this new method. The Cu complexes have good affinity for wool in neutral and weakly acid baths.

C.O.C.

Rhodamine B Equilibria

R. W. Ramette and E. B. Sandell

J. Amer. Chem. Soc., **78**, 4872-4878 (5 Oct. 1956)

Study of the benzene extraction of Rhodamine B (R) and of the solubility of its salts, and spectrophotometric examination of its aqueous solution, revealed that the colourless lactone form (R⁰) undergoes separation of charges in polar solvents to form an intensely violet zwitterion (R⁺). R⁺ can add a proton to the carboxyl group formed by the opening of the lactone ring, giving RH⁺, which has nearly the same absorption spectrum as R⁺. Addition of a second proton, probably to one of the N atoms, yields an orange species RH₂²⁺. A third proton gives RH₃³⁺, which is yellow. These colour changes are explained in terms of canonical structures. The cations can interact with anions, e.g. Cl, Br, and perchlorate, in solution to form ion-pairs having the same absorption spectra as the free cations. In addition R⁺ and the ion-pairs containing RH⁺ or RH₂²⁺ undergo dimerisation as the concentration is increased, causing apparent deviation from Beer's law.

C.O.C.

Absorption Spectra of a Methylene Blue Crystal

N. M. Melankholin

Doklady Akad. Nauk S.S.S.R., **108**, 247-250 (11 May 1956)

The author has developed a method of determining absorption spectra on single crystals ~ 20 μ. in diameter. Measurements are made on Methylene Blue in the form of microcrystals obtained by slow evaporation of a drop of soln. on a glass surface and in the form of an oriented film obtained by moving a glass rod moistened with a saturated solution of the dye in contact with a hot glass surface. Crystals oriented in a great variety of ways are examined with polarised light, the vibrations of which are adjusted so as to lie in the direction of one of the principal axes of the indicatrix. The data so obtained, together

with supplementary data obtained for the oriented film (it is shown that the molecules of the film are oriented with their planes perpendicular to the glass surface), enable absorption curves to be derived for vibrations parallel to all three axes of the indicatrix (and, for practical purposes, to the crystallographic axes). From the known arrangement of the molecules within the crystal, the absorption spectra can be related also to the axes of the Methylene Blue molecule. The absorption band found in the soln. at 656 mμ. is to be associated with the strong absorption of vibrations parallel to the long axis of the molecule, which is at a maximum in the crystal at 550 mμ. (the displacement of the band is due to intermolecular action and may be compared with the displacement produced when dimer formation occurs in soln.). Vibrations parallel to the N-S direction in the thiazine ring are absorbed only in the extreme violet (max. 405 mμ.). Vibrations perpendicular to the plane of the molecule are characterised by a single intense band at 605 mμ. Apart from these bands there is a band at 490 mμ. which is associated with a direction lying in the plane of the molecule at 45° to the shorter and longer axes; this is considered to be of intermolecular origin. All the bands are extremely sensitive to the direction of vibration of the light, being sharp for one direction only, and completely absent for a perpendicular direction.

A.E.S.

X-Ray Structure of Methylene Blue

G. S. Zhdanov, Z. V. Zronkova, and L. G. Vorontsova

Kristallografiya, **1**, 61-65 (1956);

Chem. Abs., **50**, 13549 (10 Oct. 1956)

The dye C₁₄H₁₈N₂S[Cl₄H₂O] crystallises in acicular monoclinic forms. The complex cation (C₁₄H₁₈N₂S)⁺ is planar, in typical chain elongation in the *z*-axis. The binding mechanisms between the Cl and S atoms are not covalent. The positive charges are concentrated on the S atom.

C.O.C.

N-Substituted 1-, 2-, and 4-Aminofluorene Derivatives

E. Sawicki and B. Chastain

J. Org. Chem., **21**, 1028-1030 (Sept. 1956)

Syntheses of Heterocyclic Compounds of Nitrogen. C—Photosensitising Dyes—16

T. Takahashi and K. Sato

J. Pharm. Soc. Japan, **76**, 195-198 (1956);

Chem. Abs., **50**, 13915 (10 Oct. 1956)

A series of syntheses of the following type is described. 2-Methyl-6'-Cl-pyrido[2',3':5,4]thiazole methyl iodide (0.3 g.), *p*-CH₃-CO-NH-C₆H₄-CHO (0.15) in ethyl alcohol (20 ml.), and several drops of piperidine are refluxed for 2 hr., and the product is recrystallised from methanol to yield 0.05 g. of the 2-*p*-CH₃-CO-NH-C₆H₄-CH:CH derivative.

C.O.C.

Ring Derivatives of Phenothiazine. II—2-Phenothiazinyl Ketones and their Derivatives

S. P. Massie, I. Cooke, and W. A. Hills

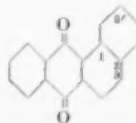
J. Org. Chem., **21**, 1006-1008 (Sept. 1956)

Acylaminoanthraquinone Vat Dyes from 1:2-Benzanthraquinonecarboxylic Acids

Z. A. Kotob

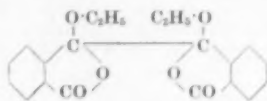
D. Tech. Sci. thesis, Swiss Federal Institute of Technology, Zürich (Prom. No. 2616, pp. 61 (1956))

The 3- and 2'-carboxylic acids of 1:2-benzanthraquinone—



were prepared by Friedel-Crafts reactions from phthalic anhydride with α - and β -methylnaphthalenes respectively, followed by oxidation of the CH₂ and cyclisation. Condensation of the corresponding carbonyl chlorides with different aminoanthraquinones gave a series of acylaminoanthraquinones, most of which, when applied as vat dyes, were weak and of poor affinity. Better were the two yellows derived from 1-amino-5-benzoylaminoanthraquinone, the two oranges from 4-amino-1:2-(*o*-chlorophenyl)anthrimidazole, and the two yellows from 4-amino-1:9-anthrapyrimidine. Fastness properties of

four of these are given but are not compared with related commercial types. In the course of confirming the structure of 2-methyl-1-naphthoyl-*o*-benzoic acid a new diethyl ester—



R.K.F.

was prepared.

Anomalous Dispersion of Light in Solutions of Inorganic Compounds

I. S. Gorban' and A. A. Shishlovskii

Doklady Akad. Nauk S.S.S.R., 108, 53-55 (1 May 1956)

Anomalous Dispersion of Light in Solutions of Dyes

Idem

Ibid., 210-213 (11 May 1956)

For various inorganic compounds the curve showing the relationship between refractive index and wavelength is accurately described by Davydov's equation (*Zhur. exp. teor. fiziki*, 24, 2, 197 (1953)), but for dyes (erythrosin, fluorescein, fuchsine, Rhodamine B) in very dil. soln. the anomalous part of the curve is often asymmetrical and so not in conformity with theory. However, when association occurs with solvent or between dye molecules (at higher concn.) the curve is of the type required by Davydov for the case of powerful molecular interaction.

A.E.S.

Barium Sulphate-Magnesia Pigments

N. R. Kamath, G. P. Kane, and P. S. Padaki

Paintindia, 6, (1), 77-82 (1956)

Chem. Abs., 50, 14241 (10 Oct. 1956)

BaS was treated with hot aq. MgSO₄ to yield a co-precipitate of BaSO₄ and Mg(OH)₂. The precipitate was heated at 840, 1060, 1150, and 1240°C. for various times, and the resulting pigments were examined. Heating for 3 hr. at 1240°C. and cooling in air gave a soft pigment of pH 9.0, alkalinity 1.8 mg. NaOH per gram, oil absorption 15.7 g./100 g., hiding power 123.4 sq.ft./lb., sp.gr. 3.994, bulk sp.gr. 1.29, n_D^{20} 1.65-1.66, and composition BaSO₄ 85.0, MgO 13.9%. Paint made from this pigment (95 parts) with TiO₂ (5) in an alkyd vehicle was as fast to weathering as a white lead-ZnO-barytes (1:1:1) linseed-oil paint.

C.O.C.

Manufacture of Pigmentary Oxides and Hydrous Oxides of Iron

Kröner

Compt. rend. 27^e Congr. intern. Chim. ind.

(Brussels), 2 (1954):

Industrie Chim. belge, 20 (Spec. No.), 595-599 (1955):

Chem. Abs., 50, 14241 (10 Oct. 1956)

Preparation, composition, colour, and crystalline shape of the oxides and hydrous oxides of iron obtained by different degrees of oxidation of iron are described.

C.O.C.

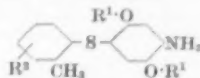
PATENTS

Diazo Components for Diazotype Printing

Ilford

BP 759,045

Diazotised amines of formula—



(R¹ = Alk of 1-3 C; R³ = H or CH₃ in 3',5', or 6' position) have a very high rate of decomposition on exposure to ultraviolet radiation and, when used in diazotype compositions, yield prints having a much clearer background than can be obtained by use of comparable compounds, e.g. the corresponding compounds which contain no CH₃ group or in which one of the CH₃ groups is *para* to the S atom.

C.O.C.

Thiazole Derivatives—Intermediates for Dyes

Kodak

BP 759,810

Compounds of formula—



(R¹ = subst. or unsubst. Alk or Ar; Q = atoms to complete a thiazolone or thiazolidone nucleus), useful as dye

intermediates, e.g. 3-carbethoxymethyl-5-(1'-ethylthio-ethylidene)-2-thiothiazolid-4-one, are obtained by condensing a compound of formula—



(R² = Alk of 1-4 C) with one of formula R³-SH.

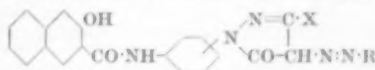
C.O.C.

Pyrazoloneazoarylides of 3:2-Hydroxynaphthoic Acid—Coupling Components for Green Azoic Dyes and Pigments

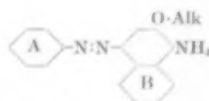
FH

BP 759,353

3:2-Hydroxynaphthoic acid is condensed with an amino-azo compound of the pyrazolone series to give aryldies—



(R = aryl free of water-solubilising groups; X = Alk or esterified COOH group), which may be coupled in substance or on the fibre with suitable diazo compounds to give pigments or azoic dyes and prints. Of particular interest are the greens from diazo compounds of aminoazo compounds—



(nuclei A and B may contain substituents, including SO₃H in B).

Thus the aryldie obtained by condensing the aminoazo compound 2-amino-5-chlorotoluene-1-*m*-aminophenyl-3-methyl-5-pyrazolone with 3:2-hydroxynaphthoic acid is dissolved in aq. alcoholic NaOH and applied to cotton. Development in a diazo soln. prepared from 1-amino-4-(2-methyl-5-nitrophenylazo)-2-ethoxynaphthalene-6-sulphonic acid and containing a little pyridine gives a green of good fastness to chlorine and to wet processing.

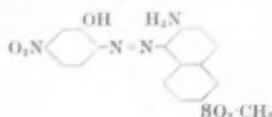
E.S.

Metal (Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

Ciba

BP 759,636

The cobalt complexes of monoazo dyes obtained by coupling a diazotised nitro-*o*-aminophenol with an *ortho*-coupling naphthylamine containing a methylsulphonyl group, both components being free from SO₃H or COOH groups, dye wool, nylon, etc. from weakly alkaline, neutral, or weakly acid baths. Thus diazotised 2-amino-5-nitrophenol is coupled under acid conditions with 6-methylsulphonyl-2-naphthylamine to give—



which is dissolved in aq. NaOH and stirred with CoSO₄ at 80-85°C. The cobalt complex so formed dyes wool green-blue.

E.S.

Acid-Base Indicator Dyes

Ciba

BP 761,044

In an azo or triphenylmethane indicator dye containing a hydrogen atom in at least one position *vicinal* to a phenolic OH group, replacement of such H atom by the group CH₂N(CH₂COOH)₂ yields derivatives which form complexes with various metal ions. In many cases formation of a metallic complex is accompanied by change of colour.

C.O.C.

Hydroxyaliphatic Esters of Phenylaminoanthraquinone Derivatives—Disperse Dyes

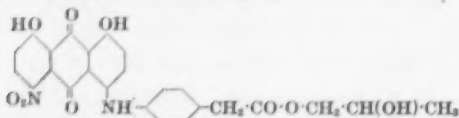
ICI

BP 756,441

α -Hydroxy- α' -nitroanthraquinones are first condensed with an amine—



(Z = CH₃, O-CH₃, C₂H₅, or C₃H₇; R = CH₃ or C₂H₅) and the resulting products treated with a glycol, which replaces R, to produce blue disperse dyes fast to burnt-gas fumes and light. Thus the dye—



is made by heating the product, obtained by condensing 1:8-dihydroxy-4:5-dinitroanthraquinone with ethyl *p*-aminophenylacetate, with propylene glycol in presence of *p*-toluenesulphonic acid at 120–130°C. for 16 hr.

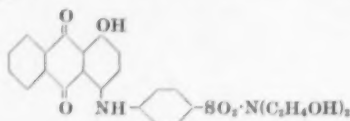
R.K.F.

Sulphamylanilinoanthraquinones—Disperse Dyes

General Aniline

BP 756,879

Anthraquinone derivatives carrying a sulphamylanilino group in one or more α -positions and H, OH, NH₂, NH-Alk, NH-Ar, or NH-CO-Alk in the other α -positions, but unsubstituted in the β -positions, are red to blue-green dyes fast to light and burnt-gas fumes. Various methods of preparation are used; e.g. an α -aminoanthraquinone is condensed with a halogenoarylsulphonamide. Thus the violet dye—



is prepared by heating at 200°C. for 5 hr. a mixture of *p*-bromo-*NN*-bis(2'-hydroxyethyl)benzenesulphonamide, 1-amino-4-hydroxyanthraquinone, sodium acetate, Na₂CO₃, cupric acetate, and nitrobenzene.

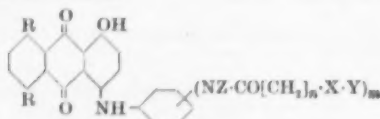
R.K.F.

Blue Disperse Dyes fast to Light and Burnt-gas Fumes

General Aniline

USP 2,723,279

Dyes of formula—



(one R = OH, the other = NO₂; X = O, S, or NZ; Y = H, CO-CH₃, Alk, hydroxyalkyl, alkoxyalkyl, hydroxyalkoxyalkyl, hydroxypolyalkoxyalkyl, or cyanoalkyl; Z = H, Alk, hydroxyalkyl, or cyanoalkyl; $n = 1-5$ $m = 1-3$) are disperse dyes which yield dyeings of good fastness to light, washing, and gas-fume fading. Thus 4:5-dinitrochrysazine, *p*-aminoglycollanilide, and *o*-dichlorobenzene are refluxed for 4 hr. and steam-distilled, and the solid product filtered off and purified by suspending for 30 min. at 90°C. in aq. sodium sesquicarbonate. The solid is filtered off, and refluxed in methanol for 30 min. On cooling, the product is filtered off and dried to yield a deep blue disperse dye.

C.O.C.

Copper Polychlorophthalocyanine Pigments

DuP

BP 757,821

The separation of copper phthalocyanine after chlorinating in an NaCl-AlCl₃ melt is done in the presence of a water-immiscible organic liquid. The resulting product is suitable for use as a pigment directly and does not require further treatment, e.g. pasting with sulphuric acid. Thus, the melt obtained by chlorinating copper phthalocyanine in NaCl-AlCl₃ is poured into cold water and cooled with ice to 35–40°C., and *o*-dichlorobenzene approx. equal in weight to the phthalocyanine added. After ca. 15-min. stirring the solvent is adsorbed on the particles of pigment, which is then filtered off and heated at 100–135°C. in *o*-dichlorobenzene. The solvent is finally distilled off in steam in presence of NaOH.

R.K.F.

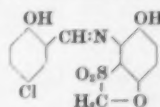
Chromium-containing Azomethin Dyes

FBY

BP 759,871

oo'-Dihydroxyazomethin dyes free from SO₃H or COOH, but containing an alkylsulphonyl group, e.g.

SO₃-CH₃ or SO₃-CH₂-O, are converted to their chromium complexes to give yellow to red dyes for wool. Thus 5-chloro-2-hydroxybenzaldehyde and aminoquinol methylene sulphone ether are heated for 30 min. at 95°C. in formamide. An aq. soln. containing K₂Cr₂O₇ and glucose is then added over 1 hr., and heating continued for 1-5 hr. at 95°C. to give the orange chromium complex of—



R.K.F.

Cobalt Phthalocyanine Intermediates

FBY

BP 759,377

The polyiminoisindolenine cobalt complexes obtained by the method of BP 687,655 (J.S.D.C., 69, 217 (1953)) are treated with aliphatic polyamines to give basic complexes suitable for application to cellulose fibre under weakly acid conditions, which on subsequent treatment with reducing agents, e.g. sodium hydrosulphite, yield blue dyeings or prints. Thus the complex obtained in Example 1 of BP 687,655 is heated in methanol containing ethylenediamine until a sample dissolves in dil. acetic acid.

R.K.F.

Phthalocyanine Pigments

American Cyanamid Co.

USP 2,723,980

Suitable phthalocyanine-forming materials are allowed to react in presence of an organic liquid diluent, a solid grinding acid added, and the pigment ground in presence of the liquid in which it was made. The product has remarkable resistance to flocculation.

USP 2,723,911

A halogen-free metal phthalocyanine, a solid grinding acid, and a liquid aromatic amine are ground, e.g. by dough grinding, the amine is extracted with a non-solvent for the pigment, and the pigment is filtered off, washed, and air-dried. The product has excellent stability, and paints containing it may be stored for long periods without any change in tinting strength.

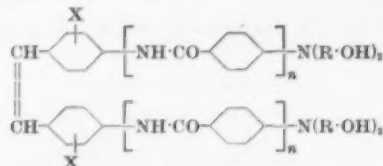
C.O.C.

Fluorescent Brightening Agents

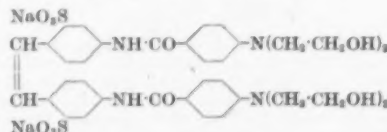
General Aniline

USP 2,723,288

Compounds of formula—



(X = SO₃H or COOH; $n = 0-4$; R = same or different alkylene of 2-4 C) are fluorescent brightening agents of excellent fastness to light and hypochlorite, which may be used in detergents without fear of building-up on repeated application. They fluoresce over a wide pH range on the alkaline and acid sides. Thus Na 4:4'-bis-(*p*-aminobenzamido)-2:2'-stilbenedisulphonate is heated in water at 60°C. with ethylene oxide, kept for 4 hr. at 60–70°C., and cooled over 12 hr. to 25°C. to yield—



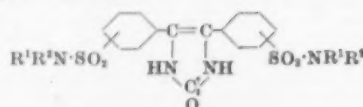
C.O.C.

Fluorescent Brightening Agents for Incorporation in Silver Halide Emulsions for Black-and-white Prints

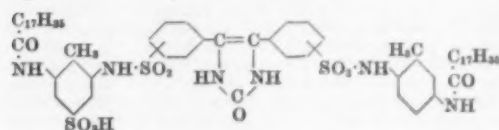
General Aniline

USP 2,723,197

Compounds of formula—



(R¹ = H or aliphatic, aromatic, or heterocyclic radical; R² = aliphatic, aromatic, or heterocyclic radical; R¹ and R² must include Alk of > 5 and a solubilising group), e.g.—



added to silver halide emulsions result in improved whites. C.O.C.

Antihalation and Filter Dyes

Agfa AG, für Photofabrikation

BP 760,739

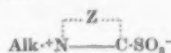
Dyes obtained by condensing *N*-alkylcarbazole-3-aldehydes with a compound containing an active methylene group or a heterocyclic N compound containing a reactive α - or γ -CH₃ group, either or both components containing at least one SO₃H or COOH group, e.g. the dye α -(*N*-ethylsulphocarbonylmethylidene)- α -benzoyl-acetonitrile, can be bleached out in normal developing solutions and have good fastness to diffusion. C.O.C.

merocyanine Dyes

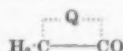
Kodak

BP 759,675

A compound of formula—



(Z = atoms to complete a pyridine or quinoline nucleus) is condensed with a compound of formula—



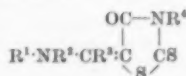
(Q = atoms to complete a thiazoline or oxazoline ring). Thus the dye 3-ethyl-5-(1-methyl-2-quinolydene)-2-thio-2:4-oxazolidione is produced by condensing the internal salt of *N*-methylquinoline-2-sulphonic acid and triethylamine with 3-ethyl-2-thio-oxazole-2:4-dione. C.O.C.

merocyanine Dyes and their Intermediates

Kodak

BP 759,323

Compounds of formula—



(R¹ and R² = H or subst. or unsubst. Alk, cycloalkyl, allyl, aralkyl, Ar, or the atoms necessary to complete a heterocyclic ring; R³ = H or Alk; R⁴ = subst. or unsubst. Alk, alkenyl, cycloalkyl, aralkyl, or Ar) on being quaternised will condense with reactive methylene compounds to give merocyanine dyes. C.O.C.

Cyanine and merocyanine Dyes

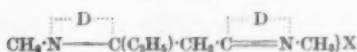
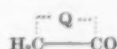
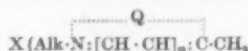
Kodak

BP 759,601

A compound of formula—



(Y = anion; Z = atoms to complete a benzothiazole or naphthothiazole nucleus), e.g. the "dimethyl sulphate of methylbenzothiazole", is condensed with one of formula—



(X = anion; n = 0 or 1; Q = atoms to complete a 5- or 6-membered heterocyclic ring; D = atoms to complete a benzothiazole, benzoxazolinazone, or naphthothiazole nucleus), e.g. 2-(2-ethyl-3-methylbenzothiazolyl)methylbenzothiazole methiodide, to yield dyes of the types 3-

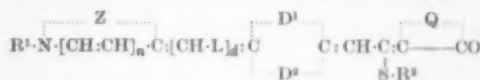
methyl-1'-ethylthia-2'-cyanine iodide, 3-carboxymethyl-5-(3-methyl-2- α -naphthothiazolylidene)rhodanine, and 3:3'-dimethyl-8-(2-ethyl-3-methyl-2- β -naphthothiazolyl)-4:5-benzo-2:2'-thiacyanine iodide. C.O.C.

Complex merocyanine Dyes having an Alkylthio or Arylthio Substituent on the Intercyclic Chain

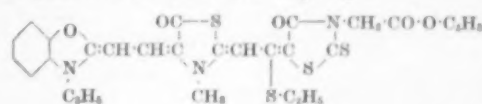
Kodak

BP 759,814

Methods are described of making dyes of formula—



(R¹ = Alk, subst. Alk, allyl, or aralkyl; R² = Alk, aralkyl, or Ar; n = 0 or 1; d = 1 or 2; L = subst. or unsubst. methin group; D¹ and D² together = atoms to complete a thiazolidone nucleus; when D¹ = S then D² = CO-NR³, and when D¹ = NR⁴ then D² = CO-S; R³ and R⁴ = Alk, subst. Alk, alkyl, or Ar; Q = atoms to complete a 2-thiothiazolid-4-one, a 2-alkylthiothiazol-5-one, or a 2-aralkylthiothiazol-5-one nucleus; Z = atoms to complete a 5- or 6-membered ring), e.g.—



C.O.C.

Caramel

Union Starch & Refining Co.

BP 759,963

Caramel of superior odour and taste and with no excessive foaming properties is obtained by maintaining the pH of the syrup during caramelisation at pH 2.6-3.8, preferably as close as possible to pH 3.0. C.O.C.

Ultramarine

American Cyanamid Co.

USP 2,723,917

Use of a mixture of air and water vapour as the oxidising gas when oxidising primary ultramarine to secondary ultramarine has none of the disadvantages that attend oxidation with SO₂. C.O.C.

Calcium Silicate Pigments

R. T. Vanderbilt Co.

BP 759,994

The gelatinised cellulosic fibrous hydrated calcium silicate pigments described in USP 2,599,094 (J.S.D.C., 69, 70 (1953)) are treated with alum so as to yield a product whose aqueous dispersion has pH 4-9. The product is especially suitable as a pigment for paper. C.O.C.

Carbon Black

Deutsche Gold- und Silber-Scheideanstalt

BP 759,919

A pigment of high wetting power and yielding oil suspensions of low viscosity is obtained by mechanically disintegrating carbon black while dispersed in at least twice its weight of hot water. C.O.C.

Pigmentary Silica

Columbia-Southern Chemical Corp.

BP 759,611

A pigment specially suitable for use in rubber is obtained by treating finely divided silica with an unsaturated silanol. C.O.C.

Rutile Titanium Dioxide

Dup

BP 760,644

Titanium tetrachloride vapour is treated with an oxygen-containing gas at 800-1350°C. in a closed reaction zone, in presence of a subchloride of Ti as a nucleating agent. C.O.C.

Nature of the Intramolecular Hydrogen Bond (VIII p. 36)

Ageing of Rubber—Dual Inhibition-Acceleration Role of Carbon Black in Rubber Oxidation (XIII p. 39)

V—PAINTS; ENAMELS; INKS

Craft and Science in Linoleum Manufacture

O. V. Soane

J. Oil & Col. Chem. Assoc., 39, 733-755 (Oct. 1956)

Mildew Problems in Surface Coatings

G. R. Lester and C. Finlay

Water and Water Eng., 60, 495-498 (Nov. 1956)

Much paint discoloration attributed to "dirt" is in fact caused by mildew. Presence of mildew can be readily

detected by use of the simple "mildewtest" kit described. Effective fungicides are now available for use in paints. C.O.C.

Lustrous and Dull Golds for Ceramics and Glass

Z. Syska

Szklo i Ceram., **7**, 79-83 (1956);

Chem. Abs., **50**, 14241 (10 Oct. 1956)

Preparation of lustrous gold paint based on Au resinate and aromatic oils is described, and recipes for 16, 20, and 20% dull red, yellow, and green gold, Ag, and Pt resinate prints are given. Syrian asphalt dissolved in $C_6H_5NO_2$ [sic] is used to increase the viscosity of the prints, rosin solution is used as thinner, and Rh, Cr, and Bi resins are added to improve the mechanical properties of the coating. C.O.C.

PATENTS

Printing Plastic Foil with Aniline and Intaglio Inks

Deutsche Gold- und Silber-Scheideanstalt BP 759,232

Adding an oxide aerogel of particle size $> 0.05 \mu$ to aniline or intaglio printing inks greatly improves their adherence to cellulose, cellulose acetate, polyethylene, or polyvinyl chloride foils. The aerogel should be formed as an aerosol by thermal decomposition of the vapour of a metal or metallised compound in presence of a hydrolysing agent. C.O.C.

White Ink for Marking Glass

Radio Corp'n. of America

USP 2,723,205

A mixture of TiO_2 (5-12 parts), eutectic lead borate (15-8) and glycerol (10-30) is applied to glass with a rubber stamp and then heated to 550°C. to yield a dense opaque print having a high degree of whiteness. C.O.C.

Non-bleeding Printing Pastes and Inks (IX p. 37)

VI—FIBRES; YARNS; FABRICS

Annual Review: Fibres

C. S. Grove, R. S. Casey, and J. L. Vodonik

Ind. Eng. Chem., **48**, 1721-1730 (Sept. Part II 1956)

A review, with 166 references (mainly 1955), covering production and consumption, new fibres, research, industrial uses, non-woven fabrics, and textile finishes. W.K.R.

Theory of Elastic Mechanisms in Fibrous Proteins

P. J. Flory

J. Amer. Chem. Soc., **78**, 5222-5235 (20 Oct. 1956)

Kinetic and Chemical Properties of Wool Cortical Cell Fractions

W. H. Ward and J. J. Bartulovich

J. Phys. Chem., **60**, 1208-1210 (Sept. 1956)

Wool cortical cells have been resolved into two main fractions of differing composition and density. Proteins made soluble by reduction of wool exposed to 6N-HCl were examined in 0.2N-NaCl + 0.5M-mercaptoethanol, at pH 8. The molecular weight of protein from both fractions is about 40,000, and the frictional ratio, calculated from the sedimentation constant and the intrinsic viscosity, is 1.8. The fraction of lower sulphur content and density is related to the "ortho" segment of the wool fibre and the heavier fraction to the "para" segment. W.K.M.

Size and Configuration of Soluble Wool Keratins

J. J. O'Donnell and E. F. Woods

J. Polymer Sci., **21**, 397-410 (Sept. 1956)

Two soluble wool proteins, S-carboxymethylkeratine-2 and α -keratin, have been studied by viscosity, sedimentation, and diffusion methods. Both proteins exist in an aggregated state in aqueous buffers. Increase of ionic strength causes increased aggregation and heterogeneity of both proteins. When stored at 25°C. both show a decrease in viscosity and for α -keratin this reflects an aggregation process. Disaggregation of both proteins may be effected by means of either 8M urea or sodium dodecyl sulphate to give a homogeneous protein of particle weight 45,000-50,000. Further reduction in molecular size can be effected with alkali at pH 13-9. Hydrodynamic parameters are calculated on the basis of a spheroidal model for the protein. W.R.M.

Sorption of Ammonia and Hydrogen Chloride by Nylon

L. H. Reyerson and L. E. Peterson

J. Phys. Chem., **60**, 1172-1176 (Sept. 1956)

Sorption isotherms for ammonia on undrawn nylon indicate that the sorption sites are carbonyl groups which are not hydrogen-bonded to imido groups. At -78°C. two molecules of HCl were taken up at very low pressure for every amide group and, at 20°C. one molecule was absorbed per amide group. Nylon fibres shrink and swell in cross-section during the early stages of HCl sorption, and slightly elongate at higher pressures. After removal of HCl the nylon showed an increase in crystallinity. It seems that HCl penetrates the nylon, breaking hydrogen bonds between adjacent chains and permitting more perfect alignment on desorption. W.R.M.

Transitions of Polyethylene Terephthalate

A. B. Thompson and D. W. Woods

Trans. Faraday Soc., **52**, 1383-1397 (Oct. 1956)

Dynamic tensile moduli and mechanical loss factors of polyethylene terephthalate in different states of orientation and crystallinity have been measured between -80° and +180°C., and from 10^{-3} to 10^4 c/s. At 1 c/s. the main transition temperature rises from 80° to 125°C. with increasing crystallinity, while its apparent activation energy falls from 182 to 97 kcal./mole. There is a second transition, at about -40°C., which is less affected by crystallinity. Possible molecular interpretations are considered. W.R.M.

PATENT

Acrylonitrile Copolymers having Improved Dyeing Properties

FH

BP 759,477

The copolymers of acrylonitrile, acrylamide or an α -substituted acrylamide, e.g. acrylo- β -dimethylaminoethylamide, containing tertiary nitrogen in the amide group, and another vinyl compound, e.g. methylvinyl sulphone, have good affinity for acid, disperse, and some vat dyes. C.O.C.

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

PATENTS

Bleaching of Cellulose Pulps

Columbia Cellulose Co.

BP 754,782

Pulp slurry having a consistency of 5-20% is boiled with 0.4-6% aq. caustic alkali at 140°-170°C. for 1-60 min. and then bleached with aq. chlorite and hypochlorite (available Cl 0.1-0.5%) for 1-6 hr. at 10-60°C. and pH 7-10. Pulps so treated not only are very pure but also have very satisfactory viscosity. K.W.

Hair Bleaching

Richard Hudnut

BP 758,762

An aq. soln. of H_2O_2 (< 10 vol.) at pH 3.5-5.5 containing a quaternary ammonium salt is applied to the hair, which is then dried without washing or rinsing. The process is repeated until the desired effect is obtained. By this method the final result obtained is the colour of the lightest natural pigment in the hair and not the artificial light straw colour usually obtained with peroxide bleaching, and there is much less damage to the hair substance. C.O.C.

Equilibrium Dialysis of Soap and Detergent Solutions (III p. 31)

VIII—DYEING

Nature of the Intramolecular Hydrogen Bond

D. N. Shigorin, M. M. Shemyakin, L. A. Shehukina, M. N. Kolosov, and F. A. Mendelevich

Doklady Akad. Nauk S.S.S.R., **108**, 672-675 (1 June 1956)

Infrared-spectrum data on O-H vibrations in 25 organic cpd. are analysed in order to determine the contributions made by π -electron interaction to the energies of intramol. hydrogen bonds in these cpd. (cf. J.S.D.C., **71**, 474 (1955)). The extent of π -electron interaction is found to depend on the presence of a unified conjugated system having an even number of members and including the ring containing the hydrogen bond together with other parts of

the molecule; it is a function of the planarity of the system and the number and distribution of the π -electrons present.

A.E.S.

Technology of Modern Oxidation Hair Dyes—I

G. S. Kass

Amer. Perfumer Aromat., **68**, (1), 25-28 (1956); *Chem. Abs.*, **50**, 13376 (25 Sept. 1956)

Hair dyes comprise (a) progressive colours, in which the desired colour is produced on the hair after several applications of the hair dye composition (the metallic dyes fall into this group), and (b) instant colours, which tint the hair when applied. The most effective preparations are the oxidation types based on *p*-phenylenediamine and related oxidation bases. Oxidation hair dye compositions are complex preparations which may contain 12-15 ingredients; they may be broken down into (i) the vehicle, (ii) the dye intermediates, and (iii) colour modifiers, stabilisers, and other assistants. The vehicle is usually an aqueous solution of soap or an anionic surfactant.

C.O.C.

PATENTS

Hair Dyeing

Société Monsavon-l'Oréal

BP 758,743

The hair is treated with a hot composition containing an organic acid and an azo or anthraquinone dye containing $\text{CO-NR}^1\text{R}^2$ or $\text{SO}_2\text{-NR}^1\text{R}^2$ (R^1 and R^2 = same or different, H, Alk, hydroxyalkyl, Ar, subst. Ar and cycloalkyl and contain at least one SO_2H or COOH group), e.g. Supraide Orange 3J (1-*p*-N-hexylacetamidophenylazo-2-hydroxy-6:8-naphthalenedisulphonic acid) (1 part) is dissolved in boiling water (100), and glacial acetic acid (3.5) added. The solution is cooled, and the condensate (8) of 1 mol. of β -naphthol with 8 mol. of ethylene oxide added followed by water (to bring to 1000). The solution padded on to dark blond hair at 45°C. imparts a striking golden lustre.

C.O.C.

Treating Hair with Fluorescent Brightening Agents

Gillette Co.

BP 759,385

Application of a fluorescent brightening agent to hair during or after treatment with a compound which breaks the disulphide bonds, but before such linkage is restored, results in the effect of the brightening agent being much faster, e.g. to washing, than if it is applied to hair whose disulphide bonds are intact.

C.O.C.

Dyeing Anodised Aluminium Golden Yellow to Dark Brown

DH

BP 761,059

Dyeings fast to light and weathering are obtained by treating first with an aqueous solution of hydrolysed collagen and then with an aqueous permanganate.

C.O.C.

The Disperse Dyes—Their Development and Application

(IV p. 31)

Sorption of Ammonia and Hydrogen Chloride by Nylon

(VI p. 36)

IX—PRINTING

Xerography

W. D. Oliphant

Research, **9**, 436-442 (Nov. 1956)

Xerography, invented by C. F. Carlson in 1937 (*USP* 2,297,619, 2,357,809), is a photographic process in which reproduction of the image is controlled by electrostatic and triboelectric phenomena. The fundamentals of the technique are outlined and its applications discussed.

C.O.C.

PATENTS

Non-bleeding Printing Pastes and Inks

National Lead Co.

BP 759,570

A colouring matter is used in conjunction with an aminoalkyl titanate or zirconate. Thus a writing ink is made up of Brilliant Benzo Blue 6BA (0.3 g.), urea (0.3 g.), thioglycol (0.3 ml.), and boiling water (2 ml.). To this solution (7 parts by wt.) is added a mixture (1) of tetrabutyl titanate (3) and triethanolamine (4). A printing paste for cotton cloth is made up of Naphtol AS Supra (0.1 g.) in a 5:3 (by wt.) mixture (2 ml.) of triethanolamine and tetraisopropyl titanate, and Cellosize WSLH (10 ml.). After application it is developed with

Fast Yellow Salt GC (0.3 g.), glacial acetic acid (2 ml.), and water (50 ml.). The resulting prints are much sharper and clearer, and bleed much less, than similar prints prepared without use of the titanate.

C.O.C.

Dyes for Photographic Sensitising

Kodak

BP 760,991

A supersensitising effect is obtained by use of a mixture of a *meso*-substituted carbocyanine dye and a benzimin-azolocarbocyanine dye.

C.O.C.

Preparation of Photographic Emulsions for Colour Photography

Kodak

BP 759,869-70

Incorporating Colour Couplers in Photographic Emulsions

Kodak

BP 759,409

Colour Photography—Preventing Formation of Colour Fog or Stain

Kodak

BP 759,000

Investigations on Colour Development. XV—Semi-quinones as Colour Developers (IV p. 32)

X—SIZING AND FINISHING

Primary Adsorbed Water in Cotton Fibres

J. E. Ayer

J. Polymer Sci., **21**, 455-462 (Sept. 1956)

Two rates of drying of fibrous materials are encountered—a constant-rate period due to evaporation of free water, and a falling-rate period due to removal of bound capillary water. A second falling-rate period, attributed to evaporation of primary adsorbed or hydrate water, is shown. Six fibre-water systems are investigated. The data obtained are plotted as drying rate against percentage of dry fibre and results expressed as the molar ratio $\text{H}_2\text{O} : \text{C}_6\text{H}_{10}\text{O}_5$ at the point of intersection of the two falling-rate lines. Values obtained agree with those obtained by other workers. Drying reduces the ability of cellulose to sorb water. Mercerisation results in increased primary water take-up, but drying prior to mercerisation reduces adsorption. The method permits a quantitative determination of primary adsorbed water.

W.R.M.

PATENTS

Modifying the Properties of Fibrous Materials

ICI

BP 760,598

The materials are treated with an organic hydrazide and HNO_3 and, preferably, with HCHO , and then dried. The process has many uses: thus viscose rayon fabric treated by it has enhanced dimensional stability, treated paper has greatly increased wet fastness, and good water-repellency may be imparted to cellulosic material. In one example, cotton cloth is impregnated with sebacinic acid dihydrazide dihydrochloride from aq. soln., dried at 50°C., treated in aq. NaNO_3 , dried at 20-30°C., glazed on a catender at 200°C., baked for 10 min. at 150°C., washed for 10 min. in 2% soap soln. at 80°C., rinsed, and dried.

C.O.C.

Permanent Setting of the Twist in Yarns containing Keratinous Fibres

Monsanto

USP 2,723,213

The twisted yarn is treated with an aqueous solution of a methylated melamine-formaldehyde precondensate, and a mercapto-aliphatic mono(or poly)hydric alcohol or a mercapto-aliphatic carboxylic acid for < 10 min., and then cured in presence or absence of a catalyst.

C.O.C.

Dimensional Stabilisation of Threads of Synthetic Organic Filaments

J. & P. Coats

BP 760,837

Treatment with an aqueous soln. or dispersion of a synthetic resin followed by subjection to dry heat yields yarns which do not shrink in subsequent dyeing.

C.O.C.

Crease-resistant Finish

Linen Industry Research Assn.

BP 758,391

Material containing cotton is impregnated with a synthetic resin precondensate, dried, and then treated with saturated steam at about 100°C. in presence of HCHO vapour with a moisture content $\geq 15\%$ by wt. of the dry untreated fabric.

C.O.C.

Flame-retardant, Water-repellent Finish for Cellulosic and Proteinaceous Textiles

DuP *USP 2,723,212*
Flammable organic material which is rapidly charred by mineral acids at temperatures below its combustion temperature is rendered flame-retardant and water-repellent by single treatment with an aqueous solution of ammonium sulphamate, a Werner-type chromium complex, dicyandiamide, and boric acid. C.O.C.

Reducing the Tendency of Animal Fibres to Felt

Commonwealth Scientific & Industrial Research Corp. *BP 758,900*
The material is impregnated with an aqueous solution or dispersion of a proteinaceous substance, which is then hardened *in situ*. Thus wool is impregnated with an aqueous dispersion of laetic casein at pH 7.5–8.0 and without drying immersed for 16 hr. in an aqueous solution of Na_2SO_4 , H_2SO_4 , and HCHO at 21°C. C.O.C.

Reducing the Felting Property of Wool

Union Carbide & Carbon Corp. *USP 2,723,924*
When wool is treated with a mildly acidic solution of resins obtained by heating aryl polyepoxides with aliphatic polyamine carboxylates, the resin is adsorbed on the wool. When the treated wool is cured, its felting properties are much reduced. C.O.C.

Rotproofing of Fibrous Materials

Ciba *BP 758,355*
The material is impregnated with an aqueous solution or emulsion of a hardenable aminoplast, there being present in either the solution or the aminoplast phase $\geq 10\%$ (on wt. of the aminoplast) of 8-hydroxyquinoline or a derivative thereof. The material is treated before, during, or after the impregnation with a water-soluble heavy-metal salt. Finally, the aminoplast is rendered insoluble in water. C.O.C.

Antistatic Finish fast to Washing and Dry-cleaning

DuP *USP 2,723,246*
The material is treated with an aqueous composition containing one part of a water-soluble polymer or copolymer of a quaternary ammonium salt of a polymerisable N-containing vinyl compound, e.g. β -acryloyloxyethyl-diethylmethylammonium methyl sulphate, and 0.2–0.8 mol. equivalent of a sulphate, sulphonate, phosphate, or monocarboxylate of $> 11\text{C}$, e.g. Na docecyl sulphate. *USP 2,723,256*

A mixture of a compound of formula—

$$[\text{CH}_2\text{-CR}^1(\text{CO-O-R}^2\text{-NR}^3\text{R}^4\text{R}^5\text{X})]_n$$
 ($\text{R}^1 = \text{H}$, CH_3 , or C_2H_5 ; $\text{R}^2 = \text{bivalent hydrocarbon radical of 1-4C}$; R^3 , R^4 , and $\text{R}^5 = \text{aliphatic hydrocarbon groups of 1-4C}$; $\text{X} = \text{hydrocarbon sulphate anion of 8-20C}$; $n = 10$), e.g. polymerised β -methacryloyloxyethyl-diethylmethylammonium hexadecenyl sulphate, and a copolymer of such a compound with $< 75\%$ of another ethylenically unsaturated monomer, e.g. vinyl acetate, is used. C.O.C.

Embossed Pile Fabric

Mohawk Carpet Mills *USP 2,723,937*
The pile is treated with a powdered binding agent and then embossed with simultaneous activation of the bonding agent in the depressed areas; e.g. a thermoplastic powder may be applied, and the fabric embossed by pressing hot elements against the areas of the pile to be depressed. C.O.C.

Annual Review: Fibres (VI p. 36)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

PATENTS

Coating Fibres in Aqueous Suspensions

F. D. Farnam Co. *BP 759,071*
An aq. slurry having a fibre content of $\geq 2\%$ is rapidly agitated in the beater by gas (air, N_2 , etc.) bubbling through it, a multivalent coagulant, e.g. $\text{Al}_2(\text{SO}_4)_3$, is added, for better retention of the coating material consisting of an emulsion of natural or synthetic rubber, thermoplastic or thermosetting resins, wax or an oil, with a solids content of 2–10%. This method prevents formation of fibre bundles or clumps of rubber, which may occur in pulp slurries of higher consistencies. K.W.

Coating of Paper

Combined Locks Paper Co. *BP 756,881*
Paper having a moisture content of 80–86% is supported on a felt and passed between two press rolls exerting a nip pressure of 41–210 lb. per linear inch, whilst a clay-containing film of sizing or coating material having a solids content of 35–50% is applied to one of the press rolls and transferred to the web, whereby any undesired pattern received from the press roll is dispersed. K.W.

Coating Aqueous Compositions on Web Supports

DuP *BP 758,401*
An aq. dispersion, e.g. of silver halides in gelatin having a viscosity of 2000–100,000 centipoises and a solids content of 20–65% is extruded from a hopper with a narrow slot orifice on to a flexible film or paper base supported on rollers and moving at a speed of at least 150 ft./min., in presence of added water vapour and the substantial absence of non-condensable gases. The coated web is passed over a large rotatable drum to a heated drying zone for 2–15 sec. at 34–150°F. and 5–192 mm. of Hg. K.W.

Modifying the Properties of Fibrous Materials (X p. 37)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Aniline Leather Finishes

A. Turow *Leather and Shoes*, 131, (21), 16–17, 21 (1956); *Chem. Abs.*, 50, 14253 (10 Oct. 1956)
The fastness to light of an aniline finish may be much poorer on leather than on wood or textiles. A properly compounded, resinated pigment base coat will act as a barrier between the leather and the aniline lacquer finish. A pigmented "clean up" coat between the base and aniline lacquer coats is often advisable. A clear lacquer top coat gives added protection. For maximum fastness an aniline lacquer made with overground pigment is preferable but is much more expensive than the usual aniline lacquer. C.O.C.

Electrophoresis of Polypeptidyl-proteins

H. V. Kley and M. A. Stahman *J. Phys. Chem.*, 60, 1200–1201 (Sept. 1956)
Changes in the electrophoretic mobility of albumen after modification by attachment of polypeptides of leucine, phenylalanine, lysine, and glutamic acid are reported. Some interpretations of the results and possible applications of polypeptidyl-proteins to physicochemical studies are discussed. W.R.M.

PATENT

Rendering Leather Water-repellent

ICI *BP 759,395*
The water-repellency of leather is much improved without adverse effect by treatment with the Werner complexes of the type described in *USP 2,273,040*, 2,356,161, and 2,524,803. C.O.C.

XIII—RUBBER; RESINS; PLASTICS

Polyester Resins—their Chemical Nature and Industrial Uses

L. H. Vaughan *Chem. and Ind.*, 996–1005 (29 Sept. 1956)
Relationship between the Viscosity of a Polymerisation System and Polymerisation Kinetics
A. N. Pravednikov

Doklady Akad. Nauk S.S.S.R., 108, 495–498 (21 May 1956)

For some monomers, e.g. styrene and vinyl acetate, the rate of mass polymerisation continuously diminishes as the reaction progresses, whereas for others, e.g. methyl acrylate and methacrylate, the reaction rate increases. The author rejects previous hypotheses advanced in explanation of this variation in behaviour. He takes the view that termination of reaction chains results mainly from reaction between polymeric radicals, and, as the activation energy of reaction-chain termination is very low, at an advanced stage of the polymerisation the process is determined essentially by the rate of diffusion of polymeric radicals toward each other, which is determined,

in its turn, by the viscosity of the medium and the mol.wt. of the polymeric radicals. Under given conditions, particularly of temperature, methyl acrylate and methacrylate give much larger polymeric radicals than styrene and vinyl acetate, but if the polymerisation is carried out at low temp. (higher viscosity and conditions favourable to the formation of larger polymeric radicals), then the latter substances should also polymerise more rapidly as reaction progresses. The author's experiments confirm that this behaviour is in fact found. A.E.S.

Flow of Polyvinyl Chloride under the Action of Large Forces

V. A. Kargin and T. I. Sogolova

Doklady Akad. Nauk S.S.S.R., 108, 662-664 (1 June 1956)

Ageing of Rubber—Dual Inhibition—Acceleration Role of Carbon Black in Rubber Oxidation

F. Lyon, K. A. Burgess, and C. W. Sweitzer

Ind. Eng. Chem., 48, 1544-1546 (Sept. 1956)

Urethane Plastics—Polymers of tomorrow

Anon.

Ind. Eng. Chem., 48, 1383-1391 (Sept. 1956)

The preparation, properties, and uses of urethane polymers are described. Their main use at present is in rigid and flexible foamed plastics, but they are likely to be used in insulating and protective coatings, adhesives, rubbers, and moulding compounds. A comparison is made of the advantages and disadvantages of urethane, rubber, vinyl, and styrene foams, and the costs of different urethane systems are discussed. Information is presented on raw-material supplies, finished-foam producers, and markets, and future trends are forecast. W.K.R.

PATENT

Improving the Surface of Thermoplastic Sheets

Celanese Corp. of America

BP 759,463

The sheets are subjected to pressure, e.g. by being drawn under tension around rollers, while immersed in molten metal. C.O.C.

XIV—ANALYSIS; TESTING; APPARATUS

Spectrophotometric Study of a Modified Molybdenum Blue Method for the Determination of Phosphorus

C. H. Lueck and D. F. Boltz

Anal. Chem., 28, 1168-1171 (July 1956)

A spectrophotometric study was made of a modified molybdenum blue method for the determination of phosphorus, in which the yellow phosphomolybdic acid is extracted with isobutyl alcohol and subsequently reduced with chlorostannous acid to molybdenum blue. The absorption spectrum of molybdenum blue in isobutyl alcohol shows characteristic absorption max. at 625 and 725 m μ . The system obeys Beer's law, with an optimum concn. of 0.1-1.3 p.p.m. of phosphorus, determined in 1-cm. cells, at 725 m μ . The effect of soln. variables is investigated; arsenic and germanium may be removed by prior volatilisation as bromides. L.T.W.

Use of Paper Chromatography for the Differential Analysis of Phosphate Mixtures

E. Karl-Kroupa

Anal. Chem., 28, 1091-1097 (July 1956)

A new quantitative method of paper chromatography has been developed for the analysis of mixtures containing ortho-, pyro-, and tri-phosphates and also cyclic (tri- and tetra-metaphosphates) and long-chain phosphates. In this method the second solvent advances in the direction opposite to that of the first. Both this method and the Ebel method, in which the solvents are run at right-angles to each other, are suitable for routine determinations in control laboratories. A special colorimetric procedure makes possible the rapid quant. evaluation of the chromatographic fractions. Procedures are described for the analysis of commercial sodium triphosphate, built detergents (no previous treatment of the sample being necessary), and surface waters. The hydrolysis which occurs during the chromatographic run has been investigated, and small correction factors are recommended for use when high accuracy is required. L.T.W.

Simultaneous Microdetermination of Copper and Iron using Mixed Phenanthrolines

B. Zak and N. Resder

Anal. Chem., 28, 1158-1161 (July 1956)

Microgram quantities of Cu and Fe may be determined simultaneously by the spectrophotometric analysis of the mixed phenanthrolines in either one or two phases. The components are determined in water or isopentyl alcohol, the result being derived by the solution of simultaneous equations, or the copper is extracted as a soln. of cuprous-neocuproine complex in isopentyl alcohol, while the ferrous-1:10-phenanthroline complex remains in the aq. phase. Accuracy is fair when both substances are in the same phase, and good for the two-phase system, in which the extracted substance has a very favourable partition coefficient. L.T.W.

Behaviour of Acid-Base Indicators in Anhydrous Acetic Acid

T. Higuchi, J. A. Feldman, and C. R. Rehm

Anal. Chem., 28, 1120-1130 (July 1956)

Results are given of spectrophotometric and potentiometric studies of anhyd. acetic acid soln. of the following indicators—Ethyl Red (C.I. 807), Pinacyanol (C.I. 808), 4-dimethylamino-4'-nitrostilbene, 9-(4-dimethylamino-benzylidene)-2-nitrofluorene, 4-dimethylamino-4'-sulphamoylazobenzene, Quinaldine Red, 5-(4-dimethylamino-benzylidene)rhodanine, NN-dimethyl-3-nitroaniline, Brilliant Cresyl Blue (C.I. 877), 1-naphtholbenzein (bis(4-hydroxy-1-naphthyl)phenylmethanol), Nile Blue A (C.I. 913), Sudan III (C.I. 248), and Sudan IV (C.I. 258). L.T.W.

Determination of Artificial Colouring Agents on Oranges and in Orange Products

S. V. Ting

Proc. Florida State Hort. Soc., 68, 157-160 (1955);

Chem. Abs., 50, 13326 (25 Sept. 1956)

Artificial colouring matter is extracted and separated from natural pigments by means of an alumina column, or it may be washed off with CHCl₃ and the concentration determined photometrically. With an absorption peak of 490 m μ , a straight-line relation between absorbance and concentration at 500 m μ was obtained. C.O.C.

Investigation of Crystals of Synthetic Ruby coloured with Chromium Sesquioxide

N. A. Toropov and I. F. Andreev

Trudy Leningrad tekhnol. Inst. Leningra.

(29), 90-98 (1954);

Chem. Abs., 50, 13395 (25 Sept. 1956)

The absorption spectrum of the ruby crystals is used to determine photometrically the amount of the colouring oxide. C.O.C.

Importance of Temperature and Relative Humidity in Light Fastness Testing

K. McLaren

J.S.D.C., 72, 527-537 (Nov. 1956)

Differences in temperature during exposure are unlikely to be a direct cause of anomalous results in light fastness testing; differences in effective humidity, however, are of extreme importance and can cause variations of as much as four grades. The effective humidity during daylight exposure in Great Britain by the method specified in B.S. 1006:1955 has an average value of 20%. Fading lamps should be operated at the same value; the effective humidity in certain lamps is too low and cannot be increased; in others it is too high when running under the makers' recommendations, but in such cases the preferred value can be easily obtained. AUTHOR

Colour Reaction of Hexuronic Acids with Anthrone

J. R. Helbert and K. D. Brown

Anal. Chem., 28, 1098-1100 (July 1956)

The influence of various experimental factors on the colour reactions of glucuronic and galacturonic acids with anthrone in 27.5 N-H₂SO₄ has been investigated. While hexoses, methylpentoses, and pentoses react with anthrone to give a blue-green colour with max. absorbance at 620-625 m μ , uronic acids produce a pink to red colour with max. absorbance at 540-550 m μ . The intensity of the anthrone-uronic acid colour increases with age, max. absorbance being attained after about 44 hr. Mixtures of glucuronic acid with glucose and of galacturonic acid with galactose obey Beer's law and behave additively in all proportions. L.T.W.

Determination of the Degree of Crystallite Orientation in Cotton Fibres by means of the Recording X-Ray Diffraction Spectrometer

J. J. Croelly, L. Segal, and H. M. Ziffo

Text. Research J., **26**, 789-795 (Oct. 1956)

A recording Geiger-counter X-ray spectrometer with rotating specimen mount has been used to measure the degree of crystallite orientation in cotton-fibre bundles in terms of the angle of half maximum intensity (50% angle) of the 002 diffraction arc of cellulose. Determinations can be made in about one-third of the time required for photographic recording. Under favourable conditions a precision of 2% can be achieved. J.C.F.

Quantitative Analysis of Fibres by Specific Gravity, especially of Mixtures of Wool and Rayon Staple—II

E. Friesser

Textil Praxis, **11**, 1009-1010 (Oct. 1956)

Differences in sp. gr. of a wide range of natural and man-made fibres are listed. On the basis of these differences a simple method is outlined for identification of fibres, particularly for separating the component fibres in unions. Various organic solvents are given for this process. B.K.

Angora Rabbit Fibres. X—Experimental Compression-meter of a Bulk of Fibres for Studying the Felting Process

S. Okajima and S. Ikeda

J. Soc. Textile Cellulose Ind. Japan, **12**, 615-620 (Sept. 1956)

When a bulk of animal fibres such as wool is compressed repeatedly, its volume decreases gradually as the result of felting. A compression-meter was prepared in order to trace this process in water and air. The apparatus has the following features—(1) the compression can be made vertically, because the compression plate is kept always horizontal by means of a special automatic control; (2) the sensitivity of the meter is 0.01 g./sq.cm.; (3) the thickness of the sample (the fibres) under no load can be measured exactly. The good reproducibility was proved in the experiments. AUTHORS

New Method of Determination of Foaming Power

J. P. Sisley and M. Loury

World Congress on Surface-active Agents, **1**, 272-277 (1954)

Determination of Detergent Foaming Power

H. Machemer and K. Heinz

World Congress on Surface-active Agents, **1**, 278-295 (1954)

Mathematical and Statistical Determination of the Detergent Power of a Surface-active Product

M. Amouroux

World Congress on Surface-active Agents, **2**, 613-615 (1954)

Colour Evaluation in the Cane Sugar Industry

V. R. Deitz

Bur. Stand. J. Res., **57**, 159-170 (Sept. 1956);
Research Paper 2706

Fluorimetric Analysis of Amino Acids

V. G. Shore and A. B. Pardoe

Anal. Chem., **28**, 1479-1481 (Sept. 1956)

After separation by paper chromatography, amino acids are semi-quantitatively determined by measurement of the intensity of the fluorescence produced by reaction with xylose. Good accuracy, within 5-10%, may often be obtained. L.T.W.

General Colour Reaction for Nitrogen Compounds

C. Menzie

Anal. Chem., **28**, 1321-1322 (Aug. 1956)

Colour reactions occur between many classes of nitrogen cpd. and *p*-dimethylaminobenzaldehyde (Ehrlich's reagent) in a mixture of toluene and ethyl alcohol containing a little conc. H_2SO_4 . The colours obtained with many amino acids, nitrogen heterocycles, and other nitrogen cpd. are tabulated. L.T.W.

PATENTS

Hardness Testing of Water

W. Boby & Co.

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Increase in hardness of water, e.g. on exhaustion of a water-softening material, is detected by injecting intermittently a small dose of soap solution into a stream of the water passing along a tube, the localised cloud of opalescence or turbidity thus formed being measured by means of a photoelectric cell system and associated light source. K.W.

Testing the Wear Resistance of Yarns

"Licencia" Találmanyokat Értékesítő Vállalat

BP 759,091

pH Meter

CFM

BP 760,551

Fatty-acid Esters of Sucrose—Methods of Preparation (III p. 30)

Primary Adsorbed Water in Cotton Fibres (X p. 37)

XV—MISCELLANEOUS

Colours of Cobalt in Glass

M. A. Aglan and H. Moore

J. Soc. Glass Technol., **39**, 381-384 r (1955);

Chem. Abs., **50**, 13389 (25 Sept. 1956)

Examination of a number of glasses ranging in colour from the full pink given by Co oxide in borie oxide glass to the full blue of high-alkali borates and alkali silicate glasses containing Co showed that in all the glasses the Co was present as Co^{2+} . The main conclusions are—(1) the pink is caused by Co^{2+} 6-co-ordinated with O; (2) the blue is caused by Co^{2+} 4-co-ordinated with O as a $(CoO_4)^{2-}$ group existing in the glass as a structure-binding unit; (3) the greens and yellows of glasses containing halogens are caused by absorption due to the free halogen in the shorter-wavelength region of the visible spectrum superposed on absorption in the longer-wavelength region due to groups of the $(CoBr_4)^{2-}$ and $(CoI_4)^{2-}$ type; (4) the blue given by acidified Co chloride soln. is caused by $(CoCl_4)^{2-}$ and is not the same as the blue given by Co in blue Co glasses. C.O.C.

Radiation Coloration in Glasses

M. Levy

J. Soc. Glass Technol., **40**, 18-24 (Feb. 1956)

Experiments on the Use of Wetting Agents for Laying Dust during Mine Excavations

J. Chappellon

World Congress on Surface-active Agents, **3**, 953-961 (1954)

PATENTS

Pigmented Dextran

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USP 2,725,303

Dry dextran is treated with 1-20% of water in a mixing machine, the amount depending on the type of machine, and then pigment is added as mixing proceeds. When the mass is homogeneous it may be used as a paste or may be dried and ground. Inorganic pigments are especially suitable for use in this process. C.O.C.

Combination Stain Fillers

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A concentrated solution of dye in a mixture of at least two glycol ethers is mixed with a neutral paste made up of an inert filler and a resin binder. Where penetration is required an organic solvent thinner may be added. C.O.C.

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Gy

BP 762,131

Adding a condensate of formaldehyde and an aromatic sulphonic acid to the sealing bath results in a bright surface which does not require polishing. C.O.C.

Colouring Non-Ferrous Metals

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BP 760,233

A non-ferrous metal and a pigment of higher m.p. than the metal are fused together and then solidified. Thus aluminium can be coloured from light grey to black by fusing it with powdered graphite or light grey to white by using TiO_2 instead of graphite. Ceramic glazing colours can similarly be used to colour metals. C.O.C.

Coloured Monocrystalline Strontium Titanate

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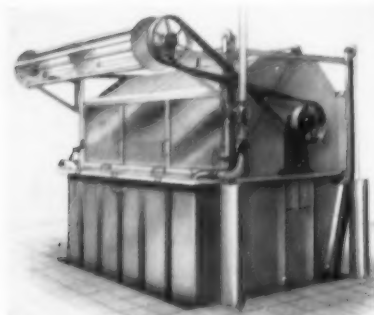
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FORTHCOMING MEETINGS OF THE SOCIETY—continued from page vi**Friday, 29th March 1957**

The Society's Annual Dinner. The Grosvenor House, Park Lane, London.

Friday, 5th April 1957

LONDON SECTION. *Some Aspects of Bleaching with Hydrogen Peroxide and Peracetic Acid.* L. Chesner, Esq., B.Sc., A.T.I. and G. C. Woodford, Esq. (Laporte Chemicals Ltd.). George Hotel, Luton. 7 p.m.

Tuesday, 9th April 1957

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by ordinary Meeting at 7.30 p.m. *Developments in Sizing as they Affect the Dyer and Finisher.* J. H. McGregor, Esq., Ph.D., F.R.I.C. and E. France, Esq., A.R.C.S., A.M.C.T. (Courtaulds Ltd.). St. Enoch Hotel, Glasgow.

Friday, 12th April 1957

MIDLANDS SECTION. Annual Dinner. George Hotel, Nottingham. 7 p.m.

Wednesday, 17th April 1957

MIDLANDS SECTION. . . . and inwardly digest (a Dissertation on Technical Literature). S. Burgess, Esq., F.S.D.C., A.M.C.T. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section). King's Head Hotel, Loughborough. 7 p.m.

Saturday, 20th April 1957

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, Bradford. 10.15 a.m.

Friday, 3rd May 1957

MIDLANDS SECTION. The Mercer Lecture of the Society. Grand Hotel, Leicester. 7 p.m.

JOURNALS WANTED

The Society is urgently wanting Journals for all months of 1955 except July and December. The months of August and September 1955 are particularly required. Copies of January, February, March, and April 1956 issues are also in demand. Please address communications to the General Secretary.



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1939

Of 1939, few people remember more than September 3rd and the beginning of World War II, but earlier in the year peaceful ventures were being made in many fields. The new Cunarder *Mauritania* made her maiden voyage to New York, the British trans-Atlantic air mail service was started with Imperial Airways *Caribou*, and nylon stockings were first sold.

Progress was not lacking in the chemical industry, and Laporte were now making hydrogen peroxide in strengths of up to 90% by weight.

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All inquiries relating to Advertisements should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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Further particulars and forms of application may be obtained from the Registrar, Nottingham and District Technical College, Shakespeare Street, Nottingham, to whom completed forms should be returned as soon as possible.

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Candidates should write giving details of experience and full personal particulars to Box V118.

ENGLISH Dyestuff Manufacturers require practical calico printer and dyer, prepared to travel abroad, and capable of demonstrating their products on the Works scale. Write in first instance giving full details of experience and salary required, etc., to Box V120.

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MISCELLANEOUS

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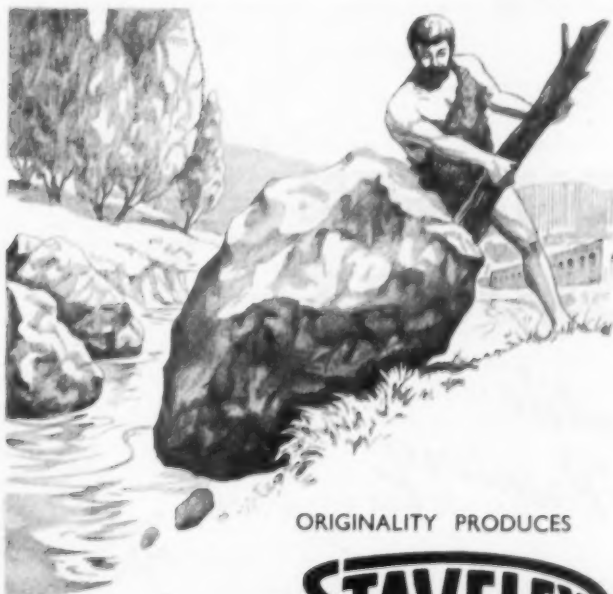
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ON

Dyeing

THE MIDLAND HOTEL MANCHESTER

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A.G.M. AND ANNUAL DINNER

★

Grosvenor House Park Lane London

Friday 29 March 1957

A.G.M. 3.45 pm

Dinner 7 for 7.45 pm

Chairmen and Secretaries Conference 2 pm

14TH MERCER LECTURE

by J. S. WARD Esq B.Sc.

ON

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